

Revised Site Investigation Work Plan

Sportsman's Park

735 South West Street

Naperville, Illinois

Prepared for:

Naperville Park District

320 West Jackson Avenue

Naperville, Illinois

January 2012

Revised Site Investigation Work Plan

***Sportsman's Park
735 South West Street
Naperville, Illinois 60540***

Shaw Project No. 144531

January 2012

Prepared for:

Naperville Park District
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1.0 INTRODUCTION

The purpose of this Revised Site Investigation Work Plan (Work Plan) is to convey the strategies and methodologies that will be used to collect and analyze soil, sediment, and groundwater samples from Sportsman's Park located at 735 South West Street, Naperville, Illinois (hereinafter referred to as the "Site"). The Site location is shown on **Figure 1**. The objective of this work plan is to convey the methods and rationale associated with performing the proposed data collection activities. Soil, sediment, surface water, and groundwater sampling methodology will be performed in a manner consistent with Illinois Environmental Protection Agency (IEPA) Site Remediation Program (SRP) regulations and supplemental guidance, as applicable.

This Work Plan is being submitted to the IEPA SRP. Shaw Environmental, Inc. (Shaw) and the Naperville Park District (NPD) desire this investigation (and future investigations and/or remediation that may be warranted) be performed in consultation with the IEPA SRP. It is the NPD's intent to ultimately receive a Focused No Further Remediation (NFR) determination for the Site, for the following constituents: Polynuclear Aromatics (PNAs), Arsenic, Barium, Cadmium, Chromium, Lead, Mercury, Selenium, Silver, Antimony, Copper, and Zinc.

The IEPA's approval of this Work Plan is requested. No fieldwork will be performed until written confirmation of the IEPA's approval is received.

1.1 Background Information

The Site is managed by the NPD and owned by the City of Naperville. The NPD leased the property in 1988 for a 99-year term. Since 1948, Sportsman's Park utilization has included gun club activities, which are conducted by the Naperville Sportsman's Club.

The Site is inclusive of approximately 27 acres of land and irregular in shape. The Site is improved with three structures. These include a club house for the Naperville Sportsman's Club, a storage facility for Sportsman's Park, and a seasonal storage facility for the NPD. The balance of the Site includes a pond, forested land, a nature trail, an access road and a parking lot. Trap shooting activities are restricted to within the fenced area. The site currently contains three trap stations. **Figure 2** illustrates the Site features.

A review of historic aerial photographs and the site reconnaissance indicate that a small arms (i.e. rifle and pistol) shooting area was located against an earthen berm on the northern side of the property. Also, the site historically hosted two additional trap stations. The earthen berm and historic trap stations are depicted on **Figure 3**. The combined shot fall trajectories of the present and historical configurations are illustrated in **Figure 4**.

The Site was previously enrolled in the SRP. The portion of the Site located outside of the Shooting Range fence was issued a *no further remediation* determination of Total Lead.

A copy of the NFR letter is included in Appendix A. Additional information regarding the historic investigation is included in the June 2010 *Phase I Environmental Site Assessment*, which has been provided to the SRP.

Discharges from the pond are regulated under National Pollutant Discharge Elimination System (NPDES) permit (IL0073253) for analysis of Lead. A copy of the NPDES permit is included in Appendix A. In compliance with the permit, preventative measures for the balance of the Sportsman's Park property were implemented including limiting access to the gun range areas, posting signage and implementing environmental best management practices at the Park, including the required use of environmentally friendly steel shot, biodegradable targets and banning Lead shot.

The Site was subject of US EPA investigation under Comprehensive Environmental Response, Compensation, and Liability Information System (CERCLIS) in 1999. The Site is not on the National Priorities List (NPL).

Several possible risks to public health and the environment are associated with the Site. A large quantity of lead (from the historic use of lead shot/bullets) and broken clay targets containing Polynuclear Aromatic Hydrocarbons (PNAs) have been deposited at the Site.

2.0 SCOPE OF WORK

2.1 Site Investigation Objectives

Shaw proposes to perform Site investigations to evaluate and characterize the Site in accordance with the requirements of the IEPA SRP with a goal of obtaining a *Focused* NFR letter for the balance of the Site areas not previously covered by the IEPA issued *Focused* NFR letter(s). It is noted that the 1998 issued IEPA NFR letters only cover portions of the adjoining property parcels and the Sportsman's Park property beyond the existing fence line boundaries, therefore, this investigation will be focused to the interior areas bounded by the existing Site fencing not currently covered by the 1998 NFR determination.

The objective of this Work Plan is to convey the methods and rationale associated with performing the proposed data collection activities.

In general, this Site Investigation will consist of the following tasks:

- Data Collection (soil, sediment, surface water and groundwater sampling);
- Analytical Testing;
- Data Evaluation; and
- Report Preparation.

Detailed discussions of these tasks are presented in the following sections of this Work Plan.

3.0 FIELD INVESTIGATIONS

3.1 Data Collection

Data collection activities will be performed by Shaw professionals who are trained in accordance with Occupational Safety and Health Administration (OSHA) standard 29 CFR 1910.120, *Hazardous Operation and Emergency Response Guidelines*. All individuals involved with these activities will be required to conform to a Site-specific *Health and Safety Plan* prepared by the Shaw Health and Safety Officer. Soil, sediment, surface water and groundwater samples will be collected using a systematic approach that will ensure the data gathered will be representative of the Site environmental conditions. All sampling activities and laboratory work will be conducted in general accordance with the U.S. Environmental Protection Agency's (EPA's) SW 846, Test Methods for Evaluation Solid Waste, and Environmental Investigations, Standard Operating Procedure and Quality Assurance Manual. Specific methods for performing field tasks are described in subsequent sections.

3.2 Sample Collection & Field Screening Procedures

Experienced Shaw environmental scientists will perform all sampling operations. The scientists will be responsible for preserving representative samples and maintaining Daily Field Reports. Examples of the field forms that will be used during this project are included in **Attachment A**.

The sampling procedures for this characterization study will include surface, near-surface and sub-surface soils, sediment, surface water and groundwater sampling collected for laboratory analysis. The protocols for this work are described in detail in the following sections and within the applicable Shaw Standard Operating Procedures (SOPs) included in **Attachment B**. **Figure 5** shows the proposed soil sampling locations, field screening locations, sediment, surface water and groundwater sampling locations.

3.2.1 Surface/Near Surface Soil Sampling

Previous investigations have indicated that the impact from historic shooting activities is located in the upper soil horizon. Accordingly, this Site Investigation will emphasize characterization of this interval.

Surface and near surface soil samples will be obtained at the trapshooting range area on both sides of the existing site channel, at the former small arms shooting range impact berm-face and floor areas, and the remaining areas on the Sportsman's Park property within the facility's fenced boundary. Grid methodology will be utilized to determine sample location. Approximately 267 soil samples will be collected from within the existing and former shooting range areas.

- Surface and near-surface soil samples will typically be collected on 100-foot spacing across a proposed grid sampling system. Proposed sample IDs, locations and analytical suites are summarized on **Table 1** and are also depicted on **Figure 5**.
- Surface/near surface soil samples will be obtained at depths representative of 0-6 inches, 6-12 inches and 12-18 inches below the existing ground surface elevations utilizing either a stainless steel sampling trowel, hand-spade, and/or hand-auger methods (refer to **Table 1**). A GeoProbe or other IEPA approved technology may be utilized to collect soil samples underneath existing pavement.
- Information on the condition of the soil in the investigation areas, moisture content (dry, moist, or wet), indications of lead and/or steel shot, bullet fragments, target debris, etc. will be recorded in the field logbook for each sampling location. Field data reporting is discussed in *Section 6.0 Field Documentation* section of this Work Plan.
- Sampling location coordinates will be recorded using a handheld GPS receiver. All positional data will be recorded in the Illinois State Plane Coordinate system.
- Upon collection, soil samples will be directly transferred into a sterile 16 ounce plastic sample bag for subsequent X-Ray Fluorescence (XRF) field screening of Total Metals concentrations within the sample (refer to *Section 3.2.2 XRF Field Screening*).
- Each bag sample will be properly labeled with a unique sample identification number (refer to *Section 3.5 Sample Identification*).
- Following XRF field screening data evaluations, 58 select soil samples collected from the trap shooting range areas will be transferred into pre-cleaned 8-oz sample containers and submitted for laboratory analysis for Total Lead, pH and moisture content at an offsite laboratory. An additional 29 soil samples will be transferred into pre-cleaned 8-oz sample containers and submitted for laboratory analysis for RCRA-8 Total Metals, Antimony, Copper and Zinc, PNAs, pH, and moisture content.
- Following XRF field screening data evaluations, 46 select soil samples collected from the former small arms shooting range berm will be transferred into pre-cleaned 8-oz sample containers and submitted for laboratory analysis for Total Lead, pH and moisture content at an offsite laboratory. An additional 10 soil samples will be transferred into pre-cleaned 8-oz sample containers and submitted for laboratory analysis for RCRA-8 Total Metals, Antimony, Copper and Zinc, PNAs, pH, and moisture content.
- Chain-of-custody forms will be completed and signed by the field representative and submitted with the samples to the analytical laboratory (refer to *Section 3.7 Chain of Custody Procedures*).

- Based upon Total Metals results, approximately 32 select samples, from various intervals, will also be analyzed for various TCLP metals.

3.2.2 XRF Field Screening

XRF measurements of various metals concentrations within the former shooting range areas will be measured primarily *ex situ* using a field portable XRF detector according to USEPA Method 6200, Field Portable XRF Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment.

Ex situ XRF analysis is a rapid testing method that can generate a large quantity of screening-level quality data over a short time period and allows field personnel to adapt the sampling strategy in reaction to XRF readings. Prior to use at the Site, a Site-specific Radiation Protection Plan (RPP) will be developed and prepared by Shaw for the XRF Spectrometer screening activities. Only certified and trained XRF operators shall be allowed to use the XRF screening instrumentation.

General Procedures for XRF Screening Analysis

- The field portable XRF detector will be operated and maintained in accordance with procedures outlined in the operator's manual accompanying the instrument, in accordance with the Shaw Standard Operating Procedures, and requirements of the Site Specific RPP.
- Calibration and standardization of the XRF detector will be performed according to the instrument manufacturer's instructions and at a frequency consistent with the manufacturer's recommendations.
- Field personnel will follow quality assurance and quality control protocols defined in EPA Method 6200 (refer to **Appendix C**), including energy calibration checks, blank samples analysis, calibration verification checks, precision sample analysis, calculation of site-specific method detection and quantitation limits.
- Applicable Total Metals concentration data will be collected for 60 seconds by the XRF operator. At 60 seconds, concentration and standard deviation measurements will be recorded in the field logbook, and in instrument memory for later downloading.
- At each sampling location, a soil sample will be placed into a bag and labeled according to sample station location and depth of sample. Ideally, soil moisture content should be between 5 and 20 percent. If sample is wet, the samples will be retained, dried and XRF analyzed back at the Shaw office, and note on the field forms.
- The XRF reading will be taken directly through the plastic sample bag.

- Information on the condition of the sample location (i.e. built-up area, visible shot and/or clay target debris, etc), and the moisture content (dry, moist, or wet) will be recorded in the field logbook for each sampling.
- Selected soil samples will be submitted for various analyses at an offsite NELAP certified laboratory (refer to *Section 5.0*).

3.2.3 Sediment Sampling

Sediment sampling is necessary because parts of the ponds, channel, and wetlands areas located on the Site are within the projected shot-fall and clay target break zones (refer to **Figures 2 through 4**).

- Approximately 12 sediment samples will be collected from the Site at select locations corresponding to the established 50-foot grid system. Sediment Sample IDs and their locations are included on **Figure 5** and summarized within **Table 1**.
- Sediment grab samples will be collected using either an Ekman Dredge “clam-type” sediment sampler and/or a “core-type” sediment sampler below any standing surface water areas. Access to the sediment sampling points may require the use of a field portable boat and/or other sampling support platform.
- Each sediment sample will be properly labeled with a unique sample identification number (refer to *Section 3.5 Sample Identification*) and submitted to the offsite analytical laboratory for analysis. Chain-of-custody forms will be completed and signed by the field representative and submitted with the samples to the analytical laboratory.
- Sediment samples will be submitted for laboratory analysis for RCRA-8 Total Metals, Antimony, Copper and Zinc, select TCLP metals (based on total results), pH, PNAs, and moisture content at an offsite laboratory.

3.2.4 Bulk Soil Samples

Following evaluation of the initial XRF screening level data, representative soil bulk samples will be collected from the trapshooting range areas and former small arms shooting range areas. Samples will be taken by hand with a shovel or other similar sampling tool. Each hand excavation will be completed to an approximate depth of 1 foot. Two (2) composite bulk samples will be collected from the trapshooting range areas exhibiting the highest XRF indicated lead impacts, and two (2) from the former small arms shooting range areas. Each composite bulk sample will be placed in a clean 5-gallon plastic container and reserved for further laboratory treatability assessment testing, if recommended.

- During the bulk sample collection activities, the field investigator will examine the berm and/or range floor excavation areas for indications of “bullet pockets” and record the observed conditions in the field logbook.

- Each composite bulk sample will be passed through a number 10 sieve to initially quantify the presents of any physical lead shot, bullet fragments, and/or shooting target debris within the impact berms. Upon bulk sample screening, any materials retained by the number 10 screen will be returned to the bulk sample.
- Each composite bulk sample will be properly labeled with a unique sample identification number (refer to *Section 3.5 Sample Identification*). Field data reporting is discussed in *Section 6.0 Field Documentation*.

3.2.5 Soil Borings & Monitoring Well Installation

Shaw proposes to advance 7 soil borings (PMW-1 through PMW-7) as illustrated on the attached **Figure 5**. The soil borings will be converted into 2-inch diameter monitoring wells (PMW-1 through PMW-7). The purpose of the soil borings will be to vertically profile the Site subsurface geologic conditions and allow the installation of groundwater monitoring wells to assess the local groundwater conditions at the Site.

At the proposed well point locations, soil sampling will be performed at 24-inch intervals until contact with the underlying shallow groundwater and/or a saturated condition is encountered. Upon contact with the water table/and or saturated zone, drilling will continue approximately 5 feet below the water table to allow the installation of a 5-foot well screen interval and riser piping well strings.

Up to 3 soil samples per soil boring location will be collected and submitted for laboratory analysis for RCRA-8 Total Metals, Antimony, Copper and Zinc, select TCLP metals (based on total results), pH, PNAs, and moisture content at an offsite laboratory.

The proposed monitoring wells will be constructed of two-inch diameter Schedule 40 PVC with a 0.01 slot screen and a stick up riser casing. Riser sections will be added as necessary to extend the well casing to approximately 3 feet above the ground surface. A sand pack will be installed in the borehole annulus to approximately 2 feet above the elevation of the top-of-screen. A granular bentonite seal will be placed, and hydrated, above the sand pack to approximately 2 feet below the ground surface. A steel, aboveground protector with a lockable, hinged lid will then be installed in concrete over the top-of-casing. The Shaw field scientist will complete a Well Construction Report for each well.

Approximately two days after installing the monitoring wells, Shaw personnel will return to the Site to develop the monitoring wells. The purpose of development is to ensure the monitoring well screens are not clogged and the wells function as intended. Development will consist of using a disposable polyethylene bailer or a submersible pump to surge and purge the well screen interval. A minimum of ten (10) well volumes will be purged from the monitoring well until the purge water is clear to the unaided eye.

Following the well development activities, the monitoring wells will be surveyed to obtain accurate hydraulic-head measurements. Field hydraulic conductivity tests (slug-tests) shall also be performed at each well point location.

3.2.6 Groundwater Sampling

Within one week but not sooner than one day after completing the monitoring well development operations, Shaw personnel will return to the Site to obtain representative groundwater samples from the 7 newly installed monitoring wells and from the 2 existing on-site monitoring wells installed by others (total of 9 samples). Prior to sampling, at each location, Shaw field scientists will use an electronic water level indicator to measure the static water level. A disposable polyethylene bailer will then be used to purge three (3) well volumes from the well. After purging, the groundwater samples will be obtained using disposable polyethylene bailers or a low-flow peristaltic pump. New sampling equipment will be used at each monitoring well location to prevent potential cross-contamination between locations. Appropriate-sized sample containers, as supplied by the contract environmental laboratory, will be filled directly from the sampling equipment.

3.2.7 Surface Water Sampling

The NPD has performed monthly sampling of the pond in accordance with the NPDES Permit since 2000. The permit requires sampling of any discharge within the first 30 minutes of a storm event for analysis of Lead and Priority Pollutant PNAs. In general, discharge events are infrequent (a total of 14 events during the 10 year reporting history) and sampling results have not indicated the presence of a permit exceedances during the ten year reporting history, with the exception of the Total Lead value concentration during the March 2010 sampling event (0.413 mg/L). It should be noted that this sample was later noted to contain solid wood fragments which the laboratory determined had biased the sample. PNA analytical results have not exceeded method detection limits during the reporting period.

Two surface water samples will be obtained from the North Pond, channel, and South Pond (total of six samples) and analyzed for the inorganic contaminants of concern: Arsenic, Barium, Cadmium, Chromium, Lead, Mercury, Selenium, Silver, Antimony, Copper, and Zinc. The surface water sample will be collected in a laboratory provided glass container and suspended solids will be allowed sufficient time to settle. The surface water sample will be decanted into a second laboratory provided container and submitted for laboratory analysis.

One surface water sample from each of the sampling locations (North Pond, channel, and South Pond) will also be submitted for analysis of PNAs (total of three samples). The samples will be immediately preserved in laboratory provided containers.

3.3 Equipment Decontamination

Prior to use at the Site and between sampling locations, all down-hole drilling and sampling equipment, including hand trowels, augers, drilling rods, and split-spoon samplers will be decontaminated. The purpose of decontamination is to prevent potential cross-contamination between sampling locations and sample intervals. Decontamination will consist of using a steam-generating pressure washer and potable water to remove soil and other debris from the drilling equipment. In addition, the split-

spoon sampler, the field scientist's logging tools, hand trowels, hand-augers, and any other sampling equipment will be washed using a scrub brush in a solution of potable water and a non-phosphate detergent. Washed equipment will be double-rinsed with distilled water and allowed to air-dry between uses.

3.4 Sample Preservation

All representative soil, sediment, surface water and groundwater samples selected for laboratory analysis will be preserved in the field, in appropriate-sized, sterile glass containers with twist-on, Teflon-lined lids, as supplied by the contract environmental laboratory. Immediately after filling, labeling and sealing the sample containers, the containers will be placed into a cooler on ice, for the duration of the daily field activities. The samples will then be transported to the Shaw offices in St. Charles, Illinois, for temporary refrigerated storage. If feasible, the samples will be delivered to the contract environmental laboratory the same day they are collected. Otherwise, Shaw will arrange to have the samples picked up and delivered to the laboratory at the next earliest opportunity.

3.5 Sample Identification

Shaw will implement a Site-specific sample identification scheme to track the project sampling activities. Each sample will be assigned a unique sample number. Soil samples will be named based on their grid and/or other selected sampling location, as illustrated in **Figure 5**. Samples will be identified using the following nomenclature:

- A sample identified as "B-1 (10-12')" will identify a soil sample from Boring 1 obtained from 10-12 feet.
- A sample identified as "MW-1" will identify a groundwater sample recovered from Monitoring Well 1.
- A sample identified as "TR-A1, TR-A2, TR-A3 ..." will identify a soil sample from the trap-shooting range area based on its location on the sampling grid. A suffix designation of (0-6 inch, 6-12 inch, or 12-18 inch, etc.), will be added to identify the representative depth interval that the sample originated from.
- A sample identified as "SAB-1, SAB-2, SAB-3 ..." will identify a soil sample from the former small arms backstop berm area, based on its location on the sampling grid. A suffix of A, B, or C will be added to identify the representative vertical high interval that the sample originated from (i.e. A = 0 to 3 feet above range floor, B = 3 to 6 feet above range floor, and C = 6 to 9 feet above range floor, etc.).
- A sample identified as "BS-1, BS-2, BS-3 ..." will identify composite bulk samples obtained from various shooting range soils.
- A sample identified as "SED-A1, SED-A2, SED-A3 ..." will identify a sediment samples collected from a corresponding grid sample location. A suffix

designation of (0-6 inch or 6-12 inch) will be added to identify the representative depth interval that the sediment originated from.

Sample containers will also be labeled with information identifying the date and time of sample collection, and the initials of the Shaw field scientist responsible for the collection.

3.6 Investigation-Derived Waste

Wastes generated during the sampling program are expected to be non-hazardous. Investigation-derived waste (IDW) generated during sampling is expected to include sampling bags, decontamination water containing residual solid materials, and used personal protective equipment (e.g., gloves, paper towels). Liquid IDW generated from decontamination and/or monitoring well developments and sampling activities will be disposed of on the surface of the site where generated. Solid IDW (e.g., used personal protective equipment) will be placed in plastic garbage bags and disposed of at the Site's solid waste collection facilities.

3.7 Chain of Custody Procedures

Every sample obtained for laboratory analysis during this project will be logged on a Chain of Custody form. The purpose of the Chain of Custody is to document the disposition of the samples from the point of collection to the point of delivery to the contract environmental laboratory. The Chain of Custody form will accompany every shipment of samples to the contract environmental laboratory and will be reproduced with each laboratory analytical report.

As a minimum, the following information will be included on the Chain of Custody form:

- Project Name and Location;
- Date/Time of Sample Collection and Delivery to Laboratory;
- Sample Description;
- Sample Matrix, e.g., soil;
- Laboratory Analytical Suite;
- Contact Information for Shaw;
- Contact Information for Laboratory;
- Signatures of Sampling Technician(s) and Laboratory Representative(s).

When a Chain of Custody form is completed, one page of the three-page carbon form is retained and placed in the Shaw project file. The other two pages of the form accompany the samples to the contract environmental laboratory. The laboratory retains one page and returns the last page with the analytical report. Upon receipt of the laboratory analytical report, the Shaw project manager will cross-check the laboratory Chain of Custody copy with the Shaw file copy. An example of the Chain of Custody form that will be used for this project is included in **Appendix A**.

4.0 ANALYTICAL TESTING PLAN

Shaw proposes to retain First Environmental Laboratories, Inc. (First Environmental), Naperville, Illinois, as the contract environmental laboratory for this project. First Environmental Laboratories is an Illinois Environmental Protection Agency Accredited Environmental Laboratory, in accordance with Title 35 of the Illinois Administrative Code, Part 186, *Accreditation of Laboratories for Drinking Water, Wastewater and Hazardous Waste Analysis*. First Environmental's Accreditation Number is #100292.

4.1 Laboratory Testing Methodologies

Laboratory analysis will be performed at the contract environmental laboratory. Selected surface soil samples, subsurface samples, and groundwater samples from the Site will be laboratory analyzed for the following potential contaminant and physical parameters:

Soil and Sediment Analytical

- Total Metals (RCRA 8-List, plus Antimony, Copper, and Zinc), via USEPA Method 6010A & 7470A
- TCLP Metals (RCRA 8-List, plus Antimony, Copper and Zinc) via USEPA Method 1311
- pH via USEPA Method 4500H+B
- PNAs via USEPA Method 8270C

Groundwater Analytical

- Total Metals (RCRA 8-List, plus Antimony, Copper, and Zinc), via USEPA Method 6010A & 7470A
- PNAs USEPA Methods 3510/8270C

Surface Water Analytical

- Total Metals (RCRA 8-List, plus Antimony, Copper, and Zinc), via USEPA Method 6010B/3010A

4.2 Laboratory Reporting

All laboratory analytical results will be reported according to EPA method protocols. Copies of the laboratory reports will be appended to the final report for the project.

5.0 FIELD DOCUMENTATION

Summaries of the daily field activities will be recorded by the Shaw field scientist in a bound, waterproof field logbook, or on Daily Field Report forms. All daily field activities will be documented in indelible ink in the logbook or on the forms, and no erasures will be made. All corrections will consist of a single line-out deletion, followed by the sampler's initials and the date. Detailed information to be recorded in the logbook or on the forms will include:

- Date of Field Activity;
- Weather Conditions;
- Project Name and Location;
- Name of Shaw Field Scientist;
- Name of Contractor Personnel, e.g., Drillers;
- Sample number (as previously described);
- Location of sample, including station name, GPS and/or grid coordinates;
- Sample type (i.e., surface soil, subsurface soil, composite, etc.);
- Sample material description;
- Description of the sample, moisture content of soil (dry, moist, or wet);
- Unique sample tag number;
- Written Summary of Daily Activities, listed in chronological order, including field observations, measurements and comments pertinent to the Site investigation goals;
- Description of any deviation from the Work Plan (as applicable); and
- Any other pertinent data or observations identified during sampling.

Copies of Chain of Custody documentation will be appended to the Daily Field Reports and maintained by the Shaw project manager in the project files. Examples of the standard Shaw field reporting forms are included in **Attachment A**.

6.0 DATA EVALUATIONS

6.1 XRF Screening Level Data

Soil XRF screening data will be evaluated by the Shaw project manager to initially define the aerial extents, both vertical and horizontal, for the primary shooting range contaminants of concern (i.e. Lead and Arsenic, etc.). The XRF screening results will be summarized in a tabular format along with the GPS positioning coordinates. Initial XRF screening data will be uploaded to SURFER[®] Version 10, a data visualization software program, to develop iso-concentration maps of both Lead and Arsenic Site impacts. Shaw's evaluation of the XRF screening level data will form the basis, in part, for the selection of representative soil samples to be submitted for Total Metals laboratory confirmation analyses, and to assess the need for additional soil sampling to delineate potential soil impacts at the Site.

6.2 Soil and Groundwater Data

Soil and groundwater sample chemical analytical results will be evaluated by the Shaw project manager to determine if residual environmental impacts are present at the Site. The analytical results will be summarized in tabular formats, and compared directly to the Tier 1 Residential Soil Remediation Objectives (SROs), the Tier 1 Construction Worker SROs, and Groundwater Remediation Objectives (GROs) as listed in Title 35 of the Illinois Administrative Act, Part 742, *Tiered Approach to Corrective Action Objectives (TACO)*. Results that exceed the *TACO* objectives (if any) will be clearly identified in the tables. Shaw's evaluation of the data will form the basis for determining the need for additional investigations and/or remedial actions. Recommendations for additional investigations and/or remedial actions will be provided in the final report.

7.0 REPORT PREPARATION

At the conclusion of the Site investigations and laboratory analyses, Shaw will prepare a *Focused Site Investigation Report (FSIR)* to document the methodologies used and the results of the investigations. The *FSIR* will provide discussions of the following:

- Project Background;
- Investigation Methodologies;
- Data Evaluation and Endangerment Assessment;
- Findings and Opinions.

Scaled Site Maps and tabulated analytical summaries will be included in the report. Copies of the Laboratory Analytical Reports will be appended to the *FSIR*.

Following IEPA submittal and review of the *FSIR* for the investigation outlined within this Revised Site Investigation Work Plan, additional sampling and/or laboratory analysis may be required to fully delineate the extent of potential contamination in both the horizontal and vertical directions.

8.0 REFERENCES

U.S. EPA, 1998: SW 846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods.

U.S. EPA, 1997: Environmental investigations, standard operating procedure and quality assurance manual.

U.S. EPA; 2005: Best Management Practices for Lead at Outdoor Shooting Ranges.

IEPA, 1997: Project Summary for Naperville Sportsman's Park.

JMS Environmental Associates, Ltd., 2008: Lead Project Report, Lead Survey, Naperville Sportsman Club.

Interstate Technology and Regulatory Council (ITRC), 2003: Characterization and Remediation of Soils at Closed Small Arms Firing Ranges.

Shaw Standard Operating Procedures (SOPs), 2006:

El-FS-001 Field Log Book

El-FS-002 Field Logsheet

El-FS-003 Chain of Custody Documentation

El-FS-005 Custody Seals

El-FS-006 Sample Labeling

El-FS-010 Sample Homogenization

El-FS-011 Compositing

El-FS-012 Shipping and Packaging of Non Hazardous Samples

El-FS-013 Packaging and Shipping of DOT/IATA-Hazardous Samples

El-FS-014 Decontamination of Contact Sampling Equipment

El-FS-020 Data Usability Review

El-FS-101 Trowel/Spoon Surface Soil Sampling

El-FS-103 Soil Sampling Using a Soil Probe or Core-Type Sampler

El-FS-123 Sediment Sampling Using a Core Sampler

El-FS-124 Sediment Sampling Using Ponar/Ekman Type Systems

El-FS-200 Screening For Metals via XRF Spectrometry.

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Figures

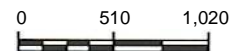


PROJECT LOCATION



Note: Figure Adapted from 2010 Google Aerial Imagery

GRAPHICAL SCALE (FT.)



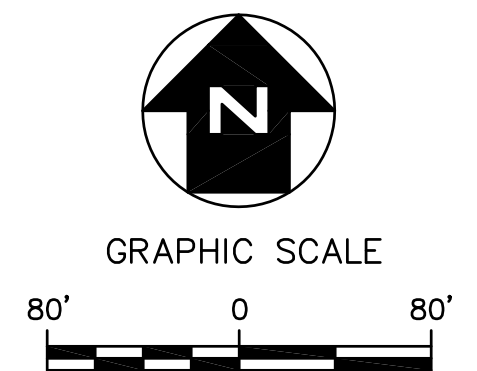
**Sportsman's Park
Naperville, Illinois**

**FIGURE 1
SITE LOCATION MAP**










APPROVED BY: EDB

PROJECT NO: 141835

DATE: June 2011

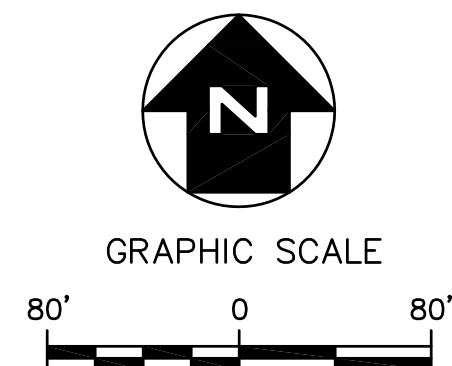


LEGEND











- | | |
|-------------------------------------------------------------------------------------|---------------------------------------------------------------|
|  | APPROXIMATE PROPERTY BOUNDARY (SPORTSMANS PARK) |
|  | APPROXIMATE ADJACENT PROPERTY BOUNDARY |
|  | CHAINLINK FENCE AND SITE REMEDIATION BOUNDARY |
|  | EXISTING MONITORING WELL |
|  | TRAP HOUSE |
|  | SHOOTING STATIONS |
|  | SHOT FALL TRAJECTORIES |
|  | ANTICIPATED SHOT FALL DEPOSITION ZONE (PRESENT CONFIGURATION) |
|  | POTENTIAL MAXIMUM SHOT FALL DISTANCE (PRESENT CONFIGURATION) |

NOTE:
CONTOUR DATA OBTAINED FROM THE ILLINOIS NATURAL RESOURCES GEOSPATIAL DATA CLEARINGHOUSE.

			<div> Shaw® Shaw Environmental, Inc.</div>	SPORTSMANS PARK NAPERVILLE, ILLINOIS	PROJ. NO.: 141835	DATE: JUNE 2011
					DESIGNED BY: JJC	DRAWING NO.
				DRAWN BY: PEL	<div>2</div> <div>2 OF 5 SHEETS</div>	
				CHECKED BY: EDB		
				APPROVED BY: DAM		
REV. NO.	DATE	DESCRIPTION			FIGURE 2 SITE FEATURES AND ANTICIPATED SHOT FALL DESPOSITION ZONE (CURRENT CONFIGURATION)	

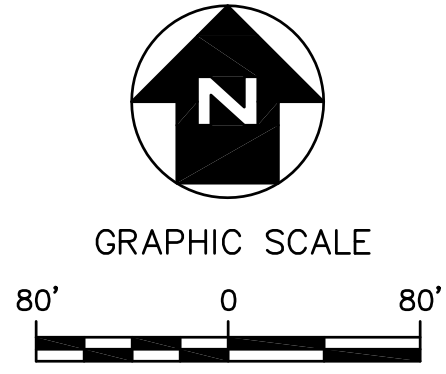


LEGEND

- | | |
|-------------------------------------------------------------------------------------|--------------------------------------------------|
|  | APPROXIMATE PROPERTY BOUNDARY (SPORTSMANS PARK) |
|  | APPROXIMATE ADJACENT PROPERTY BOUNDARY |
|  | CHAINLINK FENCE AND SITE REMEDIATION BOUNDARY |
|  | EXISTING MONITORING WELL |
|  | TRAP HOUSE |
|  | SHOOTING STATIONS |
|  | HISTORICAL SHOOTING STATIONS |
|  | HISTORICAL SHOT FALL TRAJECTORIES |
|  | HISTORICAL ANTICIPATED SHOT FALL DEPOSITION ZONE |
|  | HISTORICAL POTENTIAL MAXIMUM SHOT FALL DISTANCE |

NOTE:
CONTOUR DATA OBTAINED FROM THE ILLINOIS NATURAL RESOURCES GEOSPATIAL DATA CLEARINGHOUSE.

			 Shaw ® Shaw Environmental, Inc.	SPORTSMANS PARK NAPERVILLE, ILLINOIS	PROJ. NO.: 141835	DATE: JUNE 2011
					DESIGNED BY: JJC	DRAWING NO.
				DRAWN BY: PEL	3 3 OF 5 SHEETS	
				CHECKED BY: EDB		
				APPROVED BY: DAM		
REV. NO.	DATE	DESCRIPTION				



LEGEND

APPROXIMATE PROPERTY BOUNDARY (SPORTSMANS PARK)

APPROXIMATE ADJACENT PROPERTY BOUNDARY

CHAINLINK FENCE AND SITE REMEDIATION BOUNDARY

EXISTING MONITORING WELL

TRAP HOUSE

SHOOTING STATIONS

SHOT FALL ZONES

ANTICIPATED SHOT FALL DEPOSITION ZONE (PRESENT CONFIGURATION)

POTENTIAL MAXIMUM SHOT FALL DISTANCE (PRESENT CONFIGURATION)

HISTORICAL SHOOTING STATIONS

HISTORICAL SHOT FALL ZONES

HISTORICAL ANTICIPATED SHOT FALL DEPOSITION ZONE

HISTORICAL POTENTIAL MAXIMUM SHOT FALL DISTANCE

NOTE:
CONTOUR DATA OBTAINED FROM THE ILLINOIS NATURAL RESOURCES GEOSPATIAL DATA CLEARINGHOUSE.

REV. NO.	DATE	DESCRIPTION

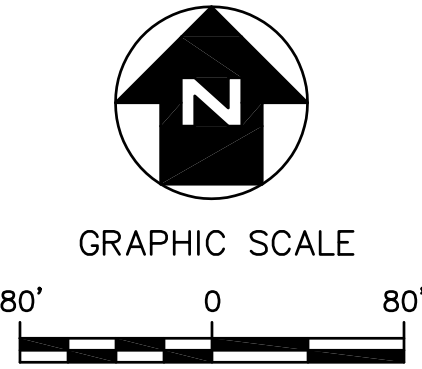
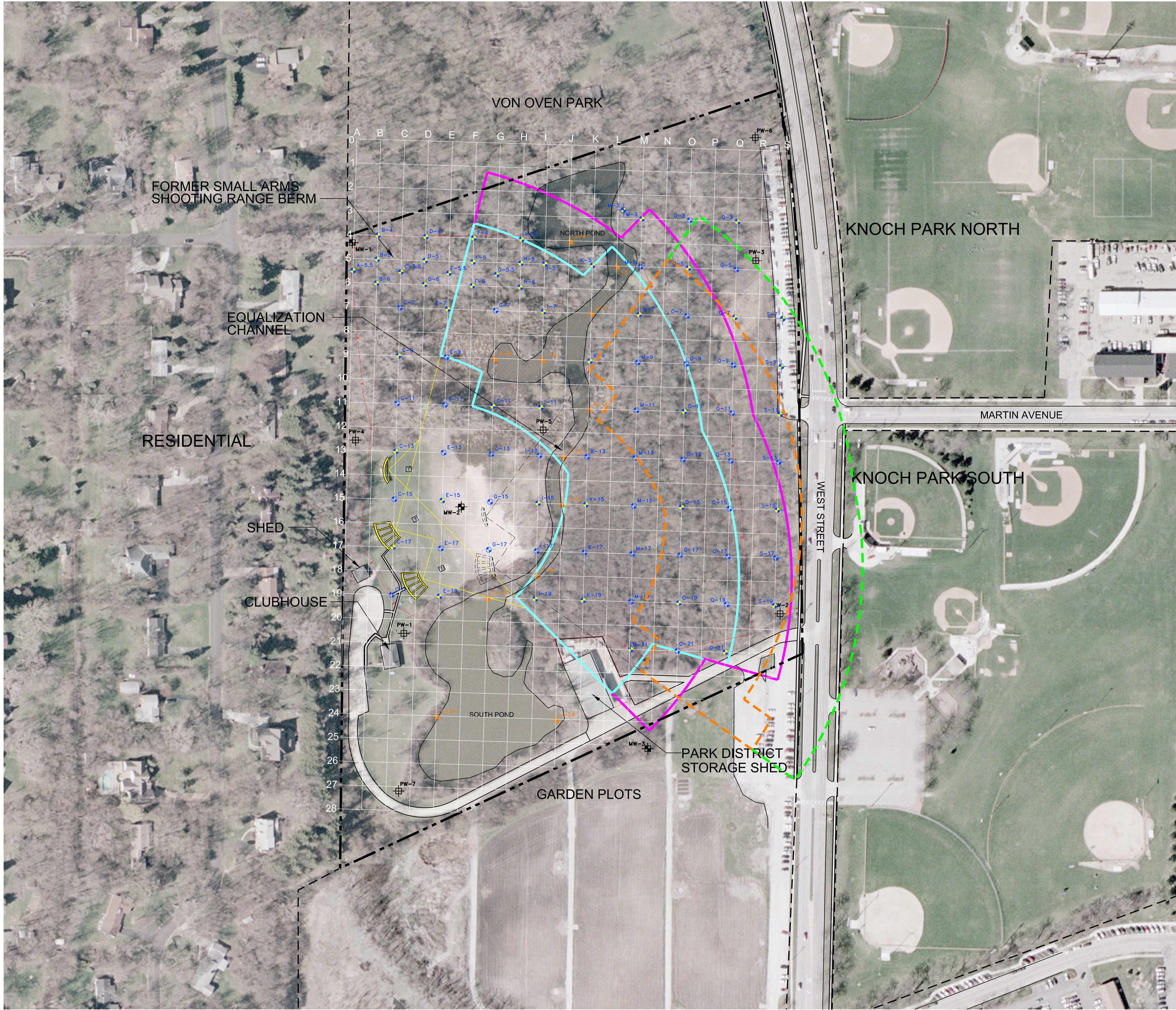


Shaw® Shaw Environmental, Inc.

SPORTSMANS PARK
NAPERVILLE, ILLINOIS

FIGURE 4
COMBINED ANTICIPATED SHOT FALL ZONES

PROJ. NO.:	141835	DATE:	JUNE 2011
DESIGNED BY:	JJC	DRAWING NO.	4
DRAWN BY:	PEL		
CHECKED BY:	EDB		
APPROVED BY:	DAM		
			4 OF 5 SHEETS



LEGEND

APPROXIMATE PROPERTY BOUNDARY (SPORTSMANS PARK)

APPROXIMATE ADJACENT PROPERTY BOUNDARY

CHAINLINK FENCE AND SITE REMEDIATION BOUNDARY

MW-1

EXISTING MONITORING WELL

T

TRAP HOUSE

SHOOTING STATIONS

SHOT FALL TRAJECTORIES

ANTICIPATED SHOT FALL DEPOSITION ZONE (PRESENT CONFIGURATION)

POTENTIAL MAXIMUM SHOT FALL DISTANCE (PRESENT CONFIGURATION)

HISTORICAL SHOOTING STATIONS

HISTORICAL SHOT FALL TRAJECTORIES

HISTORICAL ANTICIPATED SHOT FALL DEPOSITION ZONE

HISTORICAL POTENTIAL MAXIMUM SHOT FALL DISTANCE

K-9

PROPOSED XRF SOIL SAMPLE LOCATION

K-9

PROPOSED XRF AND LABORTORY ANALYSIS SOIL SAMPLE LOCATION

J-13

PROPOSED SEDIMENT SAMPLE LOCATION

MW-2

EXISTING MONITORING WELL (BY OTHERS)

PW-1

PROPOSED MONITORING WELL (BY SHAW)

A

B

0

50

1

PROPOSED SAMPLING GRID

NOTES:
1. CONTOUR DATA OBTAINED FROM THE ILLINOIS NATURAL RESOURCES GEOSPATIAL DATA CLEARINGHOUSE.
2. REVISED DECEMBER 2011.

REV. NO.	DATE	DESCRIPTION



**SPORTSMANS PARK
NAPERVILLE, ILLINOIS**

**FIGURE 5
PROPOSED SAMPLE LOCATION PLAN**

PROJ. NO.:	141835	DATE:	DECEMBER 2011
DESIGNED BY:	JJC	DRAWING NO.	
DRAWN BY:	PEL		
CHECKED BY:	EDB		
APPROVED BY:	DAM		

Tables

TABLE 1:
PROPOSED SAMPLING LOCATIONS AND ANALYTICAL SUITE
Revised December 2011

SAMPLE ID	SAMPLE DEPTH			SAMPLE ID	SAMPLE DEPTH			SAMPLE ID	SAMPLE DEPTH		
	0-6"	6-12"	12-18"		0-6"	6-12"	12-18"		0-6"	6-12"	12-18"
A-5.5	3	2	2	H-4	2	2	2	O-3	1	1	1
B-4	2	2	2	H-5	2	2	2	O-5	3	2	2
B-5	3	2	2	H-6	3	2	2	O-7	1	1	1
B-6	3	2	2	I-5*	2	2	2	O-9	1	1	1
C-5.5	3	2	2	I-7*	3	2	2	O-11	3	2	2
C-7	1	1	1	I-9*	3	-	-	O-13	1	1	1
C-9	3	2	2	I-11*	3	2	2	O-15	3	2	2
C-11	1	1	1	I-13	1	1	1	O-17	1	1	1
C-13	3	2	2	I-15	3	2	2	O-19	3	2	2
C-15	1	1	1	I-17	1	1	1	O-21	1	1	1
C-17	3	2	2	I-18*	3	-	-	Q-3	3	2	2
C-19	1	1	1	I-19*	3	2	2	Q-5	1	1	1
D-4	2	2	2	J-4	3	-	-	Q-7*	3	2	2
D-5	2	2	2	J-13*	3	-	-	Q-9*	1	1	1
D-6	3	2	2	J-15*	3	-	-	Q-11*	1	1	1
E-5.5	3	2	2	J-24	3	-	-	Q-13*	1	1	1
E-7*	3	2	2	K-5*	3	2	2	Q-15*	1	1	1
E-9*	1	1	1	K-7*	3	-	-	Q-17*	1	1	1
E-11	1	1	1	K-9*	1	1	1	Q-19*	1	1	1
E-13	1	1	1	K-11*	3	-	-	Q-21*	3	2	2
E-15	3	2	2	K-13*	1	1	1	S-7*	3	2	2
E-17	1	1	1	K-15*	3	2	2	S-9*	3	2	2
E-19	3	2	2	K-17*	1	1	1	S-11*	3	2	2
E-24	3	-	-	K-19*	3	2	2	S-13*	1	1	1
F-4*	2	2	2	L-5	3	-	-	S-15*	3	2	2
F-5*	3	2	2	M-3	3	2	2	S-17*	1	1	1
F-6*	3	2	2	M-5	1	1	1	S-19*	3	2	2
G-5.5*	3	2	2	M-7*	3	2	2	Soil Borings			
G-7*	1	1	1	M-9*	1	1	1		0-3'	3-15'	Other
G-9*	3	-	-	M-11*	1	1	1	PW-1	4	4	4
G-11*	3	2	2	M-13*	1	1	1	PW-2*	4	4	4
G-13	1	1	1	M-15*	1	1	1	PW-3	4	4	4
G-15	1	1	1	M-17*	1	1	1	PW-4	4	4	4
G-17	1	1	1	M-19*	1	1	1	PW-5*	4	4	4
G-19	3	-	-	M-21*	3	2	2	PW-6	4	4	4
								PW-7	4	4	4

Footnotes

	Small Arms Range Berm Sample
	Sediment Sample Location

- * Sample Located in Anticipated Primary Shotfall Deposition Zone (Present & Historic Configuration Combined)
- No sample collected from this interval
- 1 XRF Screening for Total Lead (no laboratory analysis)
- 2 XRF Screening / Laboratory Analysis of Total Lead, pH and moisture content and select TCLP metals (based on total results)
- 3 XRF Screening / Laboratory Analysis of RCRA-8 Total Metals, Antimony, Copper and Zinc, select TCLP metals (based on total results), pH, PNAs, and moisture
- 4 Laboratory Analysis of RCRA-8 Total Metals, Antimony, Copper and Zinc, select TCLP metals (based on total results), pH, PNAs, and moisture

Appendix A

Regulator Documentation

LAW OFFICES

BROOKS, TARULIS, SCHAFFER & TIBBLE, LLC

RICHARD J. TARULIS
DAVID N. SCHAFFER
DOUGLAS C. TIBBLE

101 NORTH WASHINGTON STREET
NAPERVILLE, ILLINOIS 60540-4511

(630) 355-2101
(630) 355-7843 FAX

GERALD J. BROOKS
(1937-2007)

DAVID G. WENTZ
OF COUNSEL

BRIAN D. MOORE

September 20, 2010

Mr. Eric Shutes
Naperville Park District
320 West Jackson Avenue
Naperville, Illinois 60540

Re: Sportsman's Park

Dear Eric:

Enclosed is correspondence received at our office regarding the above referenced matter.

Very truly yours,

**BROOKS, TARULIS, SCHAFFER &
TIBBLE, LLC**

A handwritten signature in black ink, appearing to read "Doug Tibble", written over a horizontal line.

Douglas C. Tibble

DCT/lgh
Enc.



ILLINOIS ENVIRONMENTAL PROTECTION AGENCY

1021 North Grand Avenue East, P.O. Box 19276, Springfield, Illinois 62794-9276 • (217) 782-2829
James R. Thompson Center, 100 West Randolph, Suite 11-300, Chicago, IL 60601 • (312) 814-6026

PAT QUINN, GOVERNOR

DOUGLAS P. SCOTT, DIRECTOR

217/782-0610

September 16, 2010

Naperville Park District
320 West Jackson Avenue
Naperville, Illinois 60540

Re: Naperville Park District
Sportsman's Park
NPDES Permit No. IL0073253
Final Permit

Gentlemen:

Attached is the final NPDES Permit for your discharge. The Permit as issued covers discharge limitations, monitoring, and reporting requirements. Failure to meet any portion of the Permit could result in civil and/or criminal penalties. The Illinois Environmental Protection Agency is ready and willing to assist you in interpreting any of the conditions of the Permit as they relate specifically to your discharge. The following changes have been made since the public notice of your permit:


The permittee address has been changed to the address indicated in your August 19 letter. The statement in Special Condition 4 pertaining to how to fill out the DMR form when there is no discharge has been removed, as it is duplicative to that in Special Condition 1. The date to file your DMR has been modified to the 25th of the month.

The Agency has begun a program allowing the submittal of electronic Discharge Monitoring Reports (eDMRs) instead of paper Discharge Monitoring Reports (DMRs). If you are interested in eDMRs, more information can be found on the Agency website, <http://epa.state.il.us/water/edmr/index.html>. If your facility is not registered in the eDMR program, a supply of preprinted paper DMR Forms for your facility will be sent to you prior to the initiation of DMR reporting under the reissued permit. Additional information and instructions will accompany the preprinted DMRs upon their arrival.

The attached Permit is effective as of the date indicated on the first page of the Permit. Until the effective date of any re-issued Permit, the limitations and conditions of the previously-issued Permit remain in full effect. You have the right to appeal any condition of the Permit to the Illinois Pollution Control Board within a 35 day period following the issuance date.

Should you have questions concerning the Permit, please contact Mark Liska at the 217/782-0610.

Sincerely,


Alan Keller, P.E.
Manager, Permit Section
Division of Water Pollution Control

SAK:SAK:MEL:10061603.bah

Attachment: Final Permit

cc: Records
Compliance Assurance Section
Des Plaines Region
Billing
CMAP

Rockford • 4302 N. Main St., Rockford, IL 61103 • (815) 987-7760

Elgin • 595 S. State, Elgin, IL 60123 • (847) 608-3121

Bureau of Land - Peoria • 6620 N. University St., Peoria, IL 61614 • (309) 693-5402

Collinsville • 2009 Mill Street, Collinsville, IL 62234 • (618) 346-5120

Des Plaines • 9511 W. Harrison St., Des Plaines, IL 60016 • (847) 294-4000

Peoria • 5415 N. University St., Peoria, IL 61614 • (309) 693-5463

Champaign • 2125 S. First St., Champaign, IL 61820 • (217) 278-5800

Marion • 2500 W. Main St., Suite 116, Marion, IL 62959 • (618) 993-7200

NPDES Permit No. IL0073253

Illinois Environmental Protection Agency

Division of Water Pollution Control

1021 North Grand Avenue East

Post Office Box 19276

Springfield, Illinois 62794-9276

NATIONAL POLLUTANT DISCHARGE ELIMINATION SYSTEM

Reissued (NPDES) Permit

Expiration Date: September 30, 2015

Issue Date: September 16, 2010

Effective Date: October 1, 2010

Name and Address of Permittee:

Naperville Park District
320 West Jackson Avenue
Naperville, Illinois 60540

Facility Name and Address:

Sportsman's Park
743 South West Street
Naperville, Illinois 60540
(DuPage County)

Discharge Number and Name:

001 Shooting Stations and Shot Fall Area

Receiving Waters:

Unnamed Connecting Channel Tributary to South Pond
Tributary to Unnamed Ditch Tributary to the West Branch
of the DuPage River

In compliance with the provisions of the Illinois Environmental Protection Act, Title 35 of Ill. Adm. Code, Subtitle C and/or Subtitle D, Chapter 1, and the Clean Water Act (CWA), the above-named permittee is hereby authorized to discharge at the above location to the above-named receiving stream in accordance with the standard conditions and attachments herein.

Permittee is not authorized to discharge after the above expiration date. In order to receive authorization to discharge beyond the expiration date, the permittee shall submit the proper application as required by the Illinois Environmental Protection Agency (IEPA) not later than 180 days prior to the expiration date.



Alan Keller, P.E.
Manager, Permit Section
Division of Water Pollution Control

SAK:MEL:10061603.bah

NPDES Permit No. IL0073253

Effluent Limitations and Monitoring

1. From the effective date of this permit until the expiration date, the effluent of the following discharge(s) shall be monitored and limited at all times as follows:

PARAMETER	LOAD LIMITS lbs/day <u>DAF (DMF)</u>		CONCENTRATION <u>LIMITS mg/l</u>		SAMPLE FREQUENCY	SAMPLE TYPE
	30 DAY AVERAGE	DAILY MAXIMUM	30 DAY AVERAGE	DAILY MAXIMUM		
Outfall(s): 001 - Shooting Stations and Shot Fall Area (Intermittent Discharge of Stormwater)						
Flow	See Special Condition 1					Measure When Monitoring
pH			Monitor Only		1/Month	Grab
Lead*			Monitor Only		1/Month	Grab
Priority Pollutant PNA's**			Monitor Only		1/Month	Grab

*See Special Condition 7

**See Special Condition 14

Special Conditions

SPECIAL CONDITION 1. Flow shall be reported as a daily maximum and monthly average. In the event that no discharge occurs during a given month a statement of "No Discharge" shall be reported on the DMR form for that month.

SPECIAL CONDITION 2. Samples taken in compliance with the effluent monitoring requirements shall be taken at a point representative of the discharge, but prior to entry into the unnamed ditch tributary to the West Branch of the DuPage River.

SPECIAL CONDITION 3. If an applicable effluent standard or limitation is promulgated under Sections 301(b)(2)(C) and (D), 304(b)(2), and 307(a)(2) of the Clean Water Act and that effluent standard or limitation is more stringent than any effluent limitation in the permit or controls a pollutant not limited in the NPDES Permit, the Agency shall revise or modify the permit in accordance with the more stringent standard or prohibition and shall so notify the permittee.

SPECIAL CONDITION 4. The Permittee shall record monitoring results on Discharge Monitoring Report (DMR) Forms using one such form for each outfall each month.

The Permittee may choose to submit electronic DMRs (eDMRs) instead of mailing paper DMRs to the IEPA. More information, including registration information for the eDMR program, can be obtained on the IEPA website, <http://www.epa.state.il.us/water/edmr/index.html>.

The completed Discharge Monitoring Report forms shall be submitted to IEPA no later than the 25th day of the following month, unless otherwise specified by the permitting authority.

Permittees not using eDMRs shall mail Discharge Monitoring Reports with an original signature to the IEPA at the following address:

Illinois Environmental Protection Agency
Division of Water Pollution Control
1021 North Grand Avenue East
Post Office Box 19276
Springfield, Illinois 62794-9276

SPECIAL CONDITION 5. For the purpose of this permit, this effluent is limited to storm water, free from process and other wastewater discharges.

SPECIAL CONDITION 6. The Agency may request more frequent monitoring by Letter and Without Public Notice for finite periods in the event of operational, maintenance or other problems resulting in possible effluent deterioration.

SPECIAL CONDITION 7. The water quality standards for lead at this location are 0.51 mg/l (daily maximum) and 0.107 mg/l (monthly average). Based on these standards, the Agency has developed "Action Levels" of 0.25 mg/l (daily maximum) and 0.05 mg/l (monthly average). In the event either of these "Action Levels" is exceeded on any two consecutive monthly Discharge Monitoring Reports which report an effluent, the Permittee shall initiate the development and implementation of a "Preventative Action Plan" in accordance with the compliance schedule outlined below. The Plan shall be aimed at preventing any further increase in the lead concentrations present in the effluent. (An alternative means of compliance with this requirement may include providing documentation to the Agency that the "Action Level" exceedances were not representative of the actual levels of lead in the effluent).

COMPLIANCE SCHEDULE

Item	*Compliance Date
1. Submit preliminary plan	6 months
2. Submit final plan & apply for necessary permits	12 months
3. Initiate necessary action	15 months
4. Submit an interim status report	18 months
5. Complete plan objective	24 months

*Monthly DMR forms must be submitted to the Agency no later than the 15th of the following month. Therefore, the "Compliance Date" shall be calculated from the 25th day of the month in which the DMR reporting the second "Action Level" exceedance was due.

SPECIAL CONDITION 8. Grab samples shall be collected within the first thirty minutes of effluent from the storm event.

SPECIAL CONDITION 9. The effluent, alone or in combination with other sources, shall not cause a violation of any applicable water quality standard outlined in 35 Ill. Adm. Code 302.

SPECIAL CONDITION 10. Issuance of this permit expressly prohibits the use of lead shot at this facility. Use of lead shot would require modification of this permit subject to the public notice requirements outlined in 35 Ill. Admin. Code 309. If a barrier is proposed to prevent lead shot from entering the waters of the state, design criteria must be submitted to the Agency proving that the barrier will meet

Special Conditions

this objective.

SPECIAL CONDITION 11. In the event trap-shooting activities at this facility permanently cease, the Permittee shall notify the Agency's Bureau of Land, Site Remediation Section at the address listed under Special Condition 4.

SPECIAL CONDITION 12. The permittee shall implement a Best Management Practices (BMP) program to limit and control the deposition of shotgun shell wadding, either directly or indirectly, into the waters of the State. In developing the BMP, the permittee shall evaluate the shooting activities and effluent to determine if the shell wadding is being contributed to any surface waters. If so, the BMP program shall include measures designed to control the discharge of this material into the waters of the State.

SPECIAL CONDITION 13. Issuance of this permit expressly prohibits the deposition of toxic containing target material into waters of the State.

SPECIAL CONDITION 14. Permittee shall analyze the effluent for the following polynuclear aromatic hydrocarbons (PNA's):

Acenaphthene	Acenaphthylene
Anthracene	Benzo(a)anthracene
Benzo(a)pyrene	3,4-Benzofluoranthene
Benzo(ghi)perylene	Benzo(k)fluoranthene
Chrysene	Dibenzo(a,h)anthracene
Fluoranthene	Fluorene
Indeno (1,2,3-cd)pyrene	Naphthalene
Phenanthrene	Pyrene

Priority Pollutant PNA monitoring refers to the sum of these polynuclear aromatic hydrocarbons.

ATTACHMENT H

Standard Conditions

Definitions

Act means the Illinois Environmental Protection Act, Ch. 111, 1-2 Ill. Rev. Stat. Sec. 1001-1052 as Amended.

Agency means the Illinois Environmental Protection Agency.

Board means the Illinois Pollution Control Board.

Clean Water Act (formerly referred to as the Federal Water Pollution Control Act) means Pub. L. 92-500 as amended, 33 USC 1251 et seq.

NPDES (National Pollutant Discharge Elimination System) means the national program for issuing, modifying, revoking and reissuing, terminating, monitoring and enforcing permits, and imposing and enforcing pretreatment requirements under Sections 307, 402, 318 and 405 of the Clean Water Act.

USEPA means the United States Environmental Protection Agency.

Daily Discharge means the discharge of a pollutant measured during a calendar day or any 24-hour period that reasonably represents the calendar day for purposes of sampling. For pollutants with limitations expressed in units of mass, the "daily discharge" is calculated as the total mass of the pollutant discharged over the day. For pollutants with limitations expressed in other units of measurements, the "daily discharge" is calculated as the average measurement of the pollutant over the day.

Maximum Daily Discharge Limitation (daily maximum) means the highest allowable daily discharge.

Average Monthly Discharge Limitation (30 day average) means the highest allowable average of daily discharges over a calendar month, calculated as the sum of all daily discharges measured during a calendar month divided by the number of daily discharges measured during that month.

Average Weekly Discharge Limitation (7 day average) means the highest allowable average of daily discharges over a calendar week, calculated as the sum of all daily discharges measured during a calendar week divided by the number of daily discharges measured during that week.

Best Management Practices (BMPs) means schedules of activities, prohibitions of practices, maintenance procedures, and other management practices to prevent or reduce the pollution of waters of the State. BMPs also include treatment requirements, operating procedures, and practices to control plant site runoff, spillage or leaks, sludge or waste disposal, or drainage from raw material storage.

Aliquot means a sample of specified volume used to make up a total composite sample.

Grab Sample means an individual sample of at least 100 milliliters collected at a randomly-selected time over a period not exceeding 15 minutes.

24 Hour Composite Sample means a combination of at least 8 sample aliquots of at least 100 milliliters, collected at periodic intervals during the operating hours of a facility over a 24-hour period.

8 Hour Composite Sample means a combination of at least 3 sample aliquots of at least 100 milliliters, collected at periodic intervals during the operating hours of a facility over an 8-hour period.

Flow Proportional Composite Sample means a combination of sample aliquots of at least 100 milliliters collected at periodic intervals such that either the time interval between each aliquot or the volume of each aliquot is proportional to either the stream flow at the time of sampling or the total stream flow since the collection of the previous aliquot.

- (1) **Duty to comply.** The permittee must comply with all conditions of this permit. Any permit noncompliance constitutes a violation of the Act and is grounds for enforcement action, permit termination, revocation and reissuance, modification, or for denial of a permit renewal application. The permittee shall comply with effluent standards or prohibitions established under Section 307(a) of the Clean Water Act for toxic pollutants within the time provided in the regulations that establish these standards or prohibitions, even if the permit has not yet been modified to incorporate the requirement.
- (2) **Duty to reapply.** If the permittee wishes to continue an activity regulated by this permit after the expiration date of this permit, the permittee must apply for and obtain a new permit. If the permittee submits a proper application as required by the Agency no later than 180 days prior to the expiration date, this permit shall continue in full force and effect until the final Agency decision on the application has been made.
- (3) **Need to halt or reduce activity not a defense.** It shall not be a defense for a permittee in an enforcement action that it would have been necessary to halt or reduce the permitted activity in order to maintain compliance with the conditions of this permit.
- (4) **Duty to mitigate.** The permittee shall take all reasonable steps to minimize or prevent any discharge in violation of this permit which has a reasonable likelihood of adversely affecting human health or the environment.
- (5) **Proper operation and maintenance.** The permittee shall at all times properly operate and maintain all facilities and systems of treatment and control (and related appurtenances) which are installed or used by the permittee to achieve compliance with the conditions of this permit. Proper operation and maintenance includes effective performance, adequate funding, adequate operator staffing and training, and adequate laboratory and process controls, including appropriate quality assurance procedures. This provision requires the operation of back-up, or auxiliary facilities, or similar systems only when necessary to achieve compliance with the conditions of the permit.

- (6) **Permit actions.** This permit may be modified, revoked and reissued or terminated for cause by the Agency pursuant to 40 CFR 122.62. The filing of a request by the permittee for a permit modification, revocation and reissuance, or termination or a notification of planned changes or anticipated noncompliance, does not stay any permit condition.
- (7) **Property rights.** This permit does not convey any property rights of any sort, or any exclusive privilege.
- (8) **Duty to provide information.** The permittee shall furnish to the Agency within a reasonable time any information which the Agency may request to determine whether cause exists for modifying, revoking and reissuing, or terminating this permit, or to determine compliance with the permit. The permittee shall also furnish to the Agency, upon request, copies of records required to be kept by this permit.
- (9) **Inspection and entry.** The permittee shall allow an authorized representative of the Agency, upon the presentation of credentials and other documents as may be required by law, to:
 - (a) Enter upon the permittee's premises where a regulated facility or activity is located or conducted, or where records must be kept under the conditions of this permit;
 - (b) Have access to and copy, at reasonable times, any records that must be kept under the conditions of this permit;
 - (c) Inspect at reasonable times any facilities, equipment (including monitoring and control equipment), practices, or operations regulated or required under this permit; and
 - (d) Sample or monitor at reasonable times, for the purpose of assuring permit compliance, or as otherwise authorized by the Act, any substances or parameters at any location.
- (10) **Monitoring and records.**
 - (a) Samples and measurements taken for the purpose of monitoring shall be representative of the monitored activity.
 - (b) The permittee shall retain records of all monitoring information, including all calibration and maintenance records, and all original strip chart recordings for continuous monitoring instrumentation, copies of all reports required by this permit, and records of all data used to complete the application for this permit, for a period of at least 3 years from the date of this permit, measurement, report or application. This period may be extended by request of the Agency at any time.
 - (c) Records of monitoring information shall include:
 - (1) The date, exact place and time of sampling or measurements;
 - (2) The individual(s) who performed the sampling or measurements;
 - (3) The date(s) analyses were performed;
 - (4) The individual(s) who performed the analyses;
 - (5) The analytical techniques or methods used; and
 - (6) The results of such analyses.
 - (d) Monitoring must be conducted according to test procedures approved under 40 CFR Part 136, unless other test procedures have been specified in this permit. Where no test procedure under 40 CFR Part 136 has been approved, the permittee must submit to the Agency a test method for approval. The permittee shall calibrate and perform maintenance procedures on all monitoring and analytical instrumentation at intervals to ensure accuracy of measurements.
- (11) **Signatory requirement.** All applications, reports or information submitted to the Agency shall be signed and certified.
 - (a) **Application.** All permit applications shall be signed as follows:
 - (1) For a corporation: by a principal executive officer of at least the level of vice president or a person or position having overall responsibility for environmental matters for the corporation;
 - (2) For a partnership or sole proprietorship: by a general partner or the proprietor, respectively; or
 - (3) For a municipality, State, Federal, or other public agency: by either a principal executive officer or ranking elected official.
 - (b) **Reports.** All reports required by permits, or other information requested by the Agency shall be signed by a person described in paragraph (a) or by a duly authorized representative of that person. A person is a duly authorized representative only if:
 - (1) The authorization is made in writing by a person described in paragraph (a); and
 - (2) The authorization specifies either an individual or a position responsible for the overall operation of the facility, from which the discharge originates, such as a plant manager, superintendent or person of equivalent responsibility; and
 - (3) The written authorization is submitted to the Agency.

- (c) **Changes of Authorization** If an authorization under (b) is no longer accurate because a different individual or position has responsibility for the overall operation of the facility, a new authorization satisfying the requirements of (b) must be submitted to the Agency prior to or together with any reports, information, or applications to be signed by an authorized representative.
- (12) **Reporting requirements**
- (a) **Planned changes** The permittee shall give notice to the Agency as soon as possible of any planned physical alterations or additions to the permitted facility.
- (b) **Anticipated noncompliance** The permittee shall give advance notice to the Agency of any planned changes in the permitted facility or activity which may result in noncompliance with permit requirements.
- (c) **Compliance schedules** Reports of compliance or noncompliance with or any progress reports on interim and final requirements contained in any compliance schedule of this permit shall be submitted no later than 14 days following each schedule date.
- (d) **Monitoring reports** Monitoring results shall be reported at the intervals specified elsewhere in this permit.
- (1) Monitoring results must be reported on a Discharge Monitoring Report (DMR).
- (2) If the permittee monitors any pollutant more frequently than required by the permit using test procedures approved under 40 CFR 136 or as specified in the permit, the results of this monitoring shall be included in the calculation and reporting of the data submitted in the DMR.
- (3) Calculations for all limitations which require averaging of measurements shall utilize an arithmetic mean unless otherwise specified by the Agency in the permit.
- (b) **Twenty-four hour reporting** The permittee shall report any noncompliance which may endanger health or the environment. Any information shall be provided orally within 24 hours from the time the permittee becomes aware of the circumstances. A written submission shall also be provided within 5 days of the time the permittee becomes aware of the circumstances. The written submission shall contain a description of the noncompliance and its cause, the period of noncompliance, including exact dates and times, and if the noncompliance has not been corrected, the anticipated time it is expected to continue, and steps taken or planned to reduce, eliminate, and prevent recurrence of the noncompliance. The following shall be included as information which must be reported within 24 hours:
- (1) Any unanticipated bypass which exceeds any effluent limitation in the permit.
- (2) Violation of a maximum daily discharge limitation for any of the pollutants listed by the Agency in the permit to be reported within 24 hours.
- The Agency may waive the written report on a case-by-case basis if the oral report has been received within 24 hours.
- (f) **Other noncompliance** The permittee shall report all instances of noncompliance not reported under paragraphs (12)(c), (d), or (e), at the time monitoring reports are submitted. The reports shall contain the information listed in paragraph (12)(e).
- (g) **Other information** Where the permittee becomes aware that it failed to submit any relevant facts in a permit application or submitted incorrect information in a permit application, or in any report to the Agency, it shall promptly submit such facts or information.
- (13) **Transfer of permits** A permit may be automatically transferred to a new permittee if:
- (a) The current permittee notifies the Agency at least 30 days in advance of the proposed transfer date;
- (b) The notice includes a written agreement between the existing and new permittees containing a specific date for transfer of permit responsibility coverage and liability between the current and new permittees; and
- (c) The Agency does not notify the existing permittee and the proposed new permittee of its intent to modify or revoke and reissue the permit. If this notice is not received, the transfer is effective on the date specified in the agreement.
- (14) **All manufacturing, commercial, mining, and silvicultural dischargers must notify the Agency as soon as they know or have reason to believe:**
- (a) That any activity has occurred or will occur which would result in the discharge of any toxic pollutant identified under Section 307 of the Clean Water Act which is not limited in the permit, if that discharge will exceed the highest of the following notification levels:
- (1) One hundred micrograms per liter (100 ug/l),
- (2) Two hundred micrograms per liter (200 ug/l) for acetaminophen and acrylonitrile, five hundred micrograms per liter (500 ug/l) for 2,4-dinitrophenol and for 2-methyl-4-8-dinitrophenol, and one milligram per liter (1 mg/l) for antimony.
- (3) Five (5) times the maximum concentration value reported for that pollutant in the NPDES permit application, or
- (4) The level established by the Agency in this permit.
- (b) That they have begun or expect to begin to use or manufacture as an intermediate or final product or byproduct any toxic pollutant which was not reported in the NPDES permit application.
- (15) **All Publicly Owned Treatment Works (POTWs) must provide adequate notice to the Agency of the following:**
- (a) Any new introduction of pollutants into that POTW from an indirect discharger which would be subject to Sections 301 or 306 of the Clean Water Act if it were directly discharging those pollutants; and
- (b) Any substantial change in the volume or character of pollutants being introduced into that POTW by a source introducing pollutants into the POTW at the time of issuance of the permit.
- (c) For purposes of this paragraph, adequate notice shall include information on (i) the quality and quantity of effluent introduced into the POTW, and (ii) any anticipated impact of the change on the quantity or quality of effluent to be discharged from the POTW.
- (16) **If the permit is issued to a publicly owned or publicly regulated treatment works, the permittee shall require any industrial user of such treatment works to comply with federal requirements concerning:**
- (1) User charges pursuant to Section 204(b) of the Clean Water Act, and applicable regulations appearing in 40 CFR 35.
- (2) Toxic pollutant effluent standards and pretreatment standards pursuant to Section 307 of the Clean Water Act; and
- (3) Inspection, monitoring and entry pursuant to Section 308 of the Clean Water Act.
- (17) **If an applicable standard or limitation is promulgated under Section 301(b)(2)(C) and (D), 304(b)(2), or 307(b)(2) and that effluent standard or limitation is more stringent than any effluent limitation in the permit, or controls a pollutant not limited in the permit, the permit shall be promptly modified or revoked, and reassued to conform to that effluent standard or limitation.**
- (18) **Any authorization to construct issued to the permittee pursuant to 35 Ill. Adm. Code 309.154 is hereby incorporated by reference as a condition of this permit.**
- (19) **The permittee shall not make any false statement, representation or certification in any application, record, report, plan or other document submitted to the Agency or the USEPA, or required to be maintained under this permit.**
- (20) **The Clean Water Act provides that any person who violates a permit condition implementing Sections 301, 302, 306, 307, 308, 318, or 405 of the Clean Water Act is subject to a civil penalty not to exceed \$10,000 per day of such violation. Any person who willfully or negligently violates permit conditions implementing Sections 301, 302, 306, 307, or 308 of the Clean Water Act is subject to a fine of not less than \$2,500, nor more than \$25,000 per day of violation, or by imprisonment for not more than one year, or both.**
- (21) **The Clean Water Act provides that any person who falsifies, tampers with, or knowingly renders inaccurate any monitoring device or method required to be maintained under permit shall, upon conviction, be punished by a fine of not more than \$10,000 per violation, or by imprisonment for not more than 6 months per violation, or by both.**
- (22) **The Clean Water Act provides that any person who knowingly makes any false statement, representation, or certification in any record or other document submitted or required to be maintained under this permit shall, including monitoring reports or reports of compliance or non-compliance shall, upon conviction, be punished by a fine of not more than \$10,000 per violation or by imprisonment for not more than 6 months per violation, or by both.**
- (23) **Collected screening, slimes, sludges, and other solids shall be disposed of in such a manner as to prevent entry of those wastes (or runoff from the wastes) into waters of the State. The proper authorization for such disposal shall be obtained from the Agency and is incorporated as part hereof by reference.**
- (24) **In case of conflict between these standard conditions and any other condition(s) included in this permit, the other condition(s) shall govern.**
- (25) **The permittee shall comply with, in addition to the requirements of the permit, all applicable provisions of 35 Ill. Adm. Code, Subtitle C, Subtitle D, Subtitle E, and all applicable orders of the Board.**
- (26) **The provisions of this permit are severable, and if any provision of this permit, or the application of any provision of this permit is held invalid, the remaining provisions of this permit shall continue in full force and effect.**



ILLINOIS ENVIRONMENTAL PROTECTION AGENCY

1021 North Grand Avenue East, P.O. Box 19276, Springfield, Illinois 62794-9276 Mary A. Gade, Director

217/782-6761

October 29, 1998

Mr. Peter Burchard, City Manager
City of Naperville
400 S. Eagle St.
Naperville, IL 60566

Mr. Joe Schultz, Executive Director
Naperville Park District
320 W. Jackson Avenue
Naperville, IL 60540-5275

Re: 0434675330 -- DuPage County
Naperville/City of Naperville
Site Remediation/Technical Reports

Dear Mr. Burchard:

The "Soil and Groundwater Analysis of Naperville Sportsman's Club and Adjacent Properties", (05/08/97;97-566) as prepared by Packer Engineering for the Sportsman's Park property and adjacent properties, has been reviewed by the Illinois Environmental Protection Agency (Illinois EPA). The approved remediation objectives at the remediation sites are equal to or above the existing levels of regulated substances and the Packer report shall serve as the approved Remedial Action Completion Report.

The remediation sites, consisting of approximately 41.68 acre(s) in total for the parcels investigated outside of the Sportsman's Park, are located in the City of Naperville, Illinois (see attached legal descriptions). Pursuant to Section 58.10 of the Illinois Environment Protection Act ("Act") (415 ILCS 5/58.10), your request for a no further remediation determination is granted under the conditions and terms specified in this letter.

Issuance of this focused No Further Remediation Letter ("Letter") signifies a release from further responsibilities under the Act in performing remedial actions. The Letter shall be considered prima facie evidence that the remediation sites described in the attached Site Remediation Program ("SRP") Environmental Notice and shown in the attached Site Base Map do not constitute a threat to human health and the environment for the specified recognized environmental conditions so long as the site is utilized in accordance with the terms of this Letter.

CONDITIONS AND TERMS OF APPROVAL

LEVEL OF REMEDIATION AND LAND USE LIMITATIONS

1. The recognized environmental conditions, as characterized by the investigation, consist of the following:
 - a) Regulated Substances of Concern that have been successfully addressed are detailed in the "Soil and Groundwater Analysis of Naperville Sportsman's Club and Adjacent Properties", (05/08/97;97-566)
 - b) Regulated Substances of Concern are the following:

<u>CAS No.</u>	<u>Chemical Name</u>
7439-92-1	Lead
2. In accordance with Section 58.8(c) and 58.10(b)(3) of the Act (415 ILCS 5/58.8(c) and 415 ILCS 5/58.10(b)(3), respectively), the remediation sites described in the attached SRP Environmental Notice and shown on the attached Site Base Map of this Letter is limited to residential uses.
3. The land use specified in this Letter may be revised if:
 - a) Further investigation or remedial action has been conducted that documents the attainment of objectives appropriate for the new land use; and
 - b) A new Letter is obtained and recorded in accordance with Title XVII of the Act and regulations adopted thereunder.

OTHER TERMS

4. Further information regarding this remediation site can be obtained through a written request under the Freedom of Information Act (5 ILCS 140) to:

Illinois Environmental Protection Agency
Attn: Freedom of Information Act Officer
Bureau of Land #24
1021 North Grand Avenue East
P.O. Box 19276
Springfield, IL 62794-9276
5. Pursuant to Section 58.10(f) of the Act (415 ILCS 5/58.10(f)), should the Illinois EPA

seek to void this Letter, the Illinois EPA shall provide notice to the current title holder and to the Remediation Applicant at the last known address. The notice shall specify the cause for the voidance, explain the provisions for appeal, and describe the facts in support of this cause. Specific acts or omissions that may result in the voidance of this Letter under Sections 58.10(e)(1)-(7) of the Act (415 ILCS 5/58.10(e)(1)-(7)) include, but shall not be limited to:

- a) Any violation of institutional controls or the designated land use restrictions;
 - b) The failure to operate and maintain preventive or engineering controls or to comply with any applicable groundwater monitoring plan;
 - c) If applicable, the disturbance or removal of contamination that has been left in-place in accordance with the Remedial Action Plan. Access to soil contamination may be allowed if, during and after any access, public health and the environment are protected consistent with the Remedial Action Plan;
 - d) The failure to comply with the recording requirements for this Letter;
 - e) Obtaining the Letter by fraud or misrepresentation;
 - f) Subsequent discovery of contaminants, not identified as part of the investigative or remedial activities upon which the issuance of the Letter was based, that pose a threat to human health or the environment;
 - g) The failure to pay the No Further Remediation Assessment Fee within 45 days after receiving a request for payment from the Illinois EPA;
 - h) The failure to pay in full the applicable fees under the Review and Evaluation Services Agreement within 45 days after receiving a request for payment from the Illinois EPA.
6. Pursuant to Section 58.10(d) of the Act, the No Further Remediation Letter shall apply in favor of the following persons:
- a) The City of Naperville;
 - b) The owner (City of Naperville) and operator (Naperville Park District) of the remediation site;
 - c) Any parent corporation or subsidiary of the owner of the remediation site;
 - d) Any co-owner, either by joint-tenancy, right of survivorship, or any other party

sharing a relationship with the owner of the remediation site;

- e) Any holder of a beneficial interest of a land trust or inter vivos trust, whether revocable or irrevocable, involving the remediation site;
 - f) Any mortgagee or trustee of a deed of trust of the owner of the remediation site or any assignee, transferee, or any successor-in-interest of the owner of the remediation site;
 - g) Any successor-in-interest of the owner of the remediation site;
 - h) Any transferee of the owner of the remediation site whether the transfer was by sale, bankruptcy proceeding, partition, dissolution of marriage, settlement or adjudication of any civil action, charitable gift, or bequest;
 - i) Any heir or devisee of the owner of the remediation site;
 - j) Any financial institution, as that term is defined in Section 2 of the Illinois Banking Act and to include the Illinois Housing Development Authority, that has acquired the ownership, operation, management, or control of the remediation site through foreclosure or under the terms of a security interest held by the financial institution, under the terms of an extension of credit made by the financial institution, or any successor in interest thereto; or
 - k) In the case of a fiduciary (other than a land trustee), the estate, trust estate, or other interest in property held in a fiduciary capacity, and a trustee, executor, administrator, guardian, receiver, conservator, or other person who holds the remediated site in a fiduciary capacity, or a transferee of such party.
7. This Letter, including all attachments, must be recorded as a single instrument within 45 days of receipt with the Office of the Recorder of DuPage County. For recording purposes, the SRP Environmental Notice attached to this Letter should be the first page of the instrument filed. This Letter shall not be effective until officially recorded by the Office of the Recorder of DuPage County in accordance with Illinois law so that it forms a permanent part of the chain of title for the properties investigated outside of the Sportsman's Park (see attached legal descriptions).

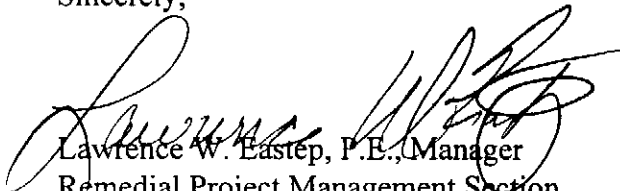
Within 30 days of this Letter being recorded by the Office of the Recorder of DuPage County, a certified copy of this Letter, as recorded, shall be obtained and submitted to the Illinois EPA to:

Mr. Robert E. O'Hara
Illinois Environmental Protection Agency
Bureau of Land/RPMS Section
1021 North Grand Avenue, East
P.O. Box 19276
Springfield, IL 62794-9276

8. In accordance with Section 58.10(g) of the Act, a No Further Remediation Assessment Fee based on the costs incurred for the remediation site by the Illinois EPA for review and evaluation services will be applied in addition to the fees applicable under the Review and Evaluation Services Agreement. Request for payment of the No Further Remediation Assessment Fee will be included with the final billing statement.

If you have any questions regarding this correspondence, you may contact the Illinois EPA project manager, Stan Komperda at 217/782-5504.

Sincerely,



Lawrence W. Eastep, P.E., Manager
Remedial Project Management Section
Division of Remediation Management
Bureau of Land

Enclosures: SRP Environmental Notice
Site Base Maps

cc: Bob O'Hara
Tim Milbrandt

PREPARED BY:

Name: Peter Burchard, City Manager

Address: City of Naperville
400 S. Eagle St.
Naperville, IL 60566

RETURN TO:

Name: Peter Burchard, City Manager

Address: City of Naperville
400 S. Eagle St.
Naperville, IL 60566

THE ABOVE SPACE FOR RECORDER'S OFFICE

This Environmental No Further Remediation letter must be submitted by the remediation applicant within 45 days of its receipt, to the Office of the Recorder of DuPage County.

Illinois State EPA Number: 0434670024

The Naperville Park District, the Remediation Applicant, whose address is 320 W. Jackson, Naperville, IL has performed investigative activities for the remediation sites depicted on the attached Site Base Map and which can be identified by the following:

1. Legal description or Reference to a Plat Showing the Boundaries:

GARDEN PLOTS: Does not include those areas within the fenced portion of the Sportsman's Park;

Plat of Survey of that part of the Southwest Quarter of Section 24, Township 38 North, Range 9 East of the third Principal Meridian described by commencing at the Southeast corner of said Southwest Quarter; thence North along the East line of said Southwest quarter a distance of 921.5 feet; thence Westerly a distance of 232.85 feet to the Southwest corner of the tract of land recorded as "West Street", according to the plat thereof recorded January 18, 1962 as document number R62-1737, for a point of beginning, thence Westerly along a line which forms an angle of 91 degrees, 37 minutes, 30 seconds measured North to West with the West line of West Street a distance of 1106.00 feet to a point on the Southerly extension of a easterly line of Charles Shuffler's Sons' Green Acres; thence Northerly along said Easterly line of Green Acres and the Southerly extension thereof a distance of 94.92 feet to the Northeast corner of Lot 14 in said Green Acres; thence Northeasterly along the Southeasterly line of Lot 13 in said Green Acres and said line, extended a distance of 1260.16 feet to a point on the West line of West Street aforesaid; thence Southerly along said West line of West Street a distance of 1488.0 feet to the point beginning, in DuPage County, Illinois. (Garden Plots/33.8 acres)

VON OVEN PROPERTY: That part of Section 24, Township 38 North, Range 9 East of the Third Principal Meridian, Described as follows:

Beginning at the point of intersection of the southerly line of Oswego Road and the east line of Shiffler's Subdivision (Green Acres); thence in an Easterly direction along the Southerly line of Oswego Road 570 feet; thence South 100 feet; thence in a Northeasterly direction 198 feet; thence South 543.18 feet; thence in a

Southwesterly direction 801.9 feet; thence North 577.5 feet along east line of Shiffler's Subdivision to the point of beginning, in the City of Naperville, DuPage County, Illinois. (7.88 acres)

2. Common Address: N/A
3. Remediation Site Owner: City of Naperville
4. Land Use Limitation: Residential
5. Site Investigation: Focused

See NFR letter for other terms.

**PROPERTY OWNER CERTIFICATION OF NFR LETTER
UNDER THE SITE REMEDIATION PROGRAM**

If the Remediation Applicant is not the sole owner of the remediation site, include the full legal name, title, the company, the street address, the city, the state, the ZIP code, and the telephone number of all other property owners. Include the site name, street address, city, ZIP code, county, Illinois Inventory I.D. number and real estate tax index/parcel index number. The property owner(s), or the duly authorized agent of the owner(s) must certify, by original signature, the statement appearing below.

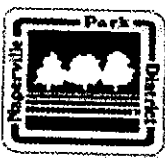
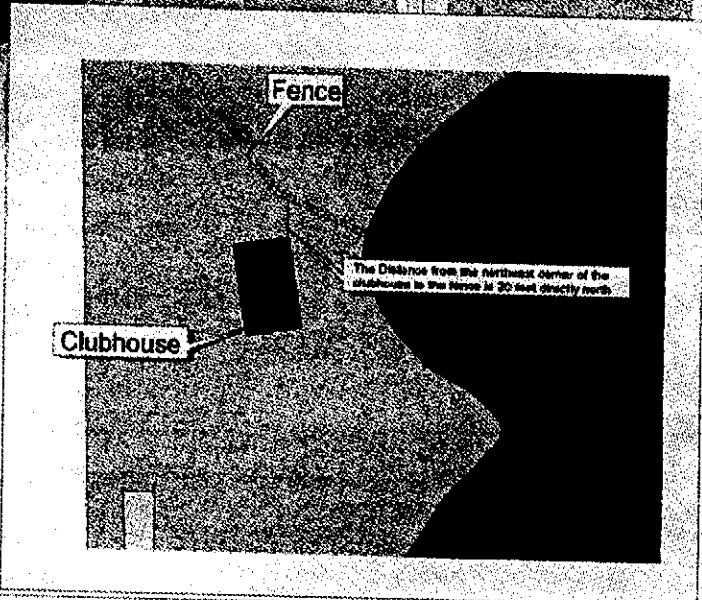
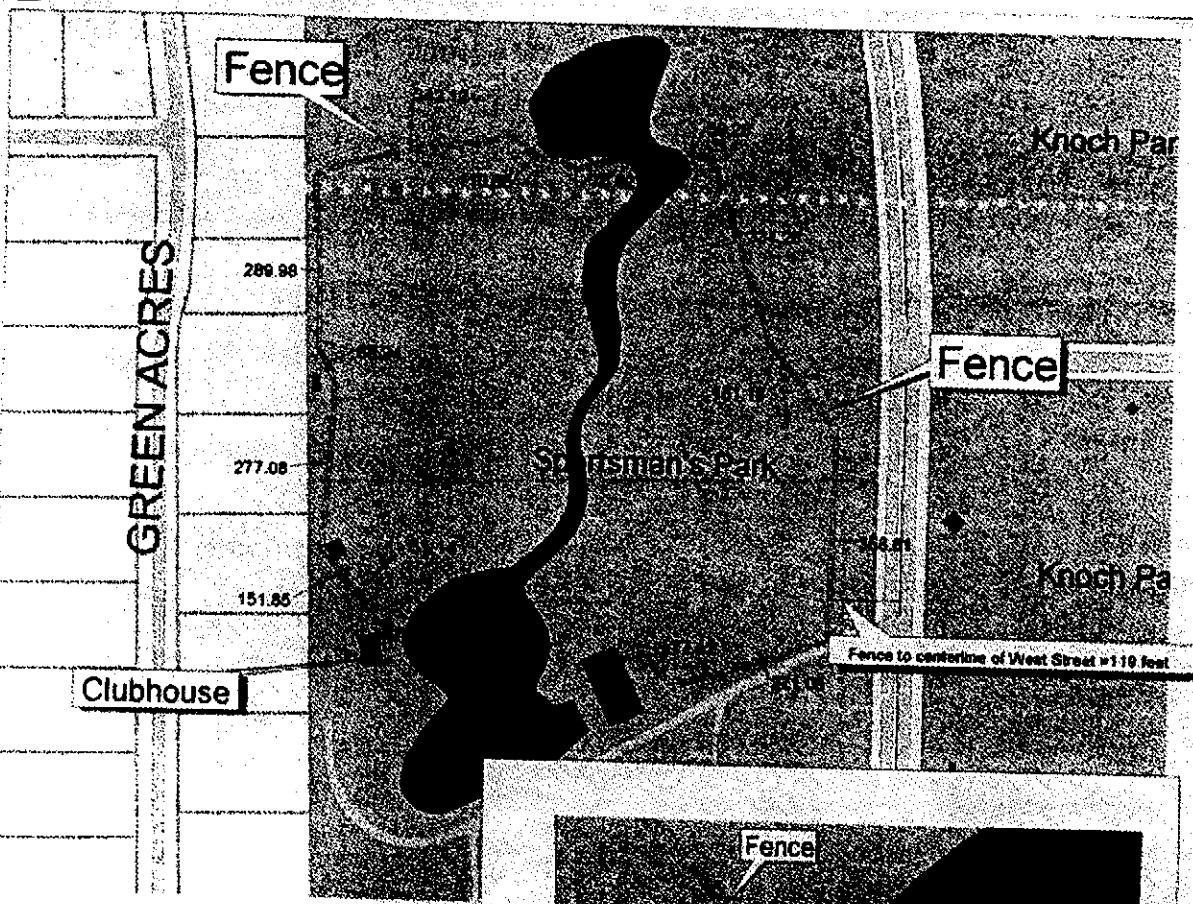
A duly authorized agent means a person who is authorized by written consent or by law to act on behalf of a property owner including, but not limited to:

1. For corporations, a principal executive officer of at least the level of vice-president;
2. For a sole proprietorship or partnership, the proprietor or a general partner, respectively; and
3. For a municipality, state or other public agency, the head of the agency or ranking elected official.

For multiple property owners, attach additional sheets containing the information described above, along with a signed, dated certification for each. All property owner certifications must be recorded along with the attached NFR letter.

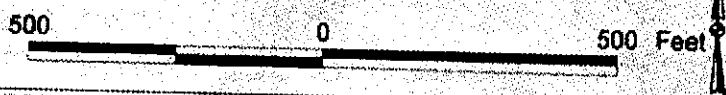
Property Owner Information	
Owner's Name: _____	
Title: _____	
Company: _____	
Street Address: _____	
City: _____	State: _____ Zip Code: _____ Phone: _____
Site Information	
Site Name: _____	
Site Address: _____	
City: _____	State: _____ Zip Code: _____ County: _____
Illinois Inventory ID Number: _____	
Real Estate Tax Index/Parcel Index No. _____	
I hereby certify that I have reviewed the attached No Further Remediation Letter, and that I accept the terms and conditions and any land use limitations set forth in the letter.	
Owner's Signature: _____ Date: _____	
SUBSCRIBED AND SWORN TO BEFORE ME this _____ day of _____, 19__	
_____ Notary Public	

The Illinois EPA is authorized to require this information under Sections 415 ILCS 5/58 - 58.12 of the Environmental Protection Act and regulations promulgated thereunder. If the Remediation Applicant is not also the sole owner of the remediation site, this form must be completed by all owners of the remediation site and recorded with the NFR Letter. Failure to do so may void the NFR Letter. This form has been approved by the Forms Management Center. All information submitted to the Site Remediation Program is available to the public except when specifically designated by the Remediation Applicant to be treated confidentially as a trade secret or secret process in accordance with the Illinois Compiled Statutes, Section 7(a) of the Environmental Protection Act, applicable Rules and Regulations of the Illinois Pollution Control Board and applicable Illinois EPA rules and guidelines.



Sportsman's Fence Dimensions

Naperville Park District
Planning Team
April 8, 1998

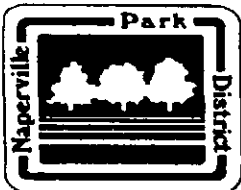
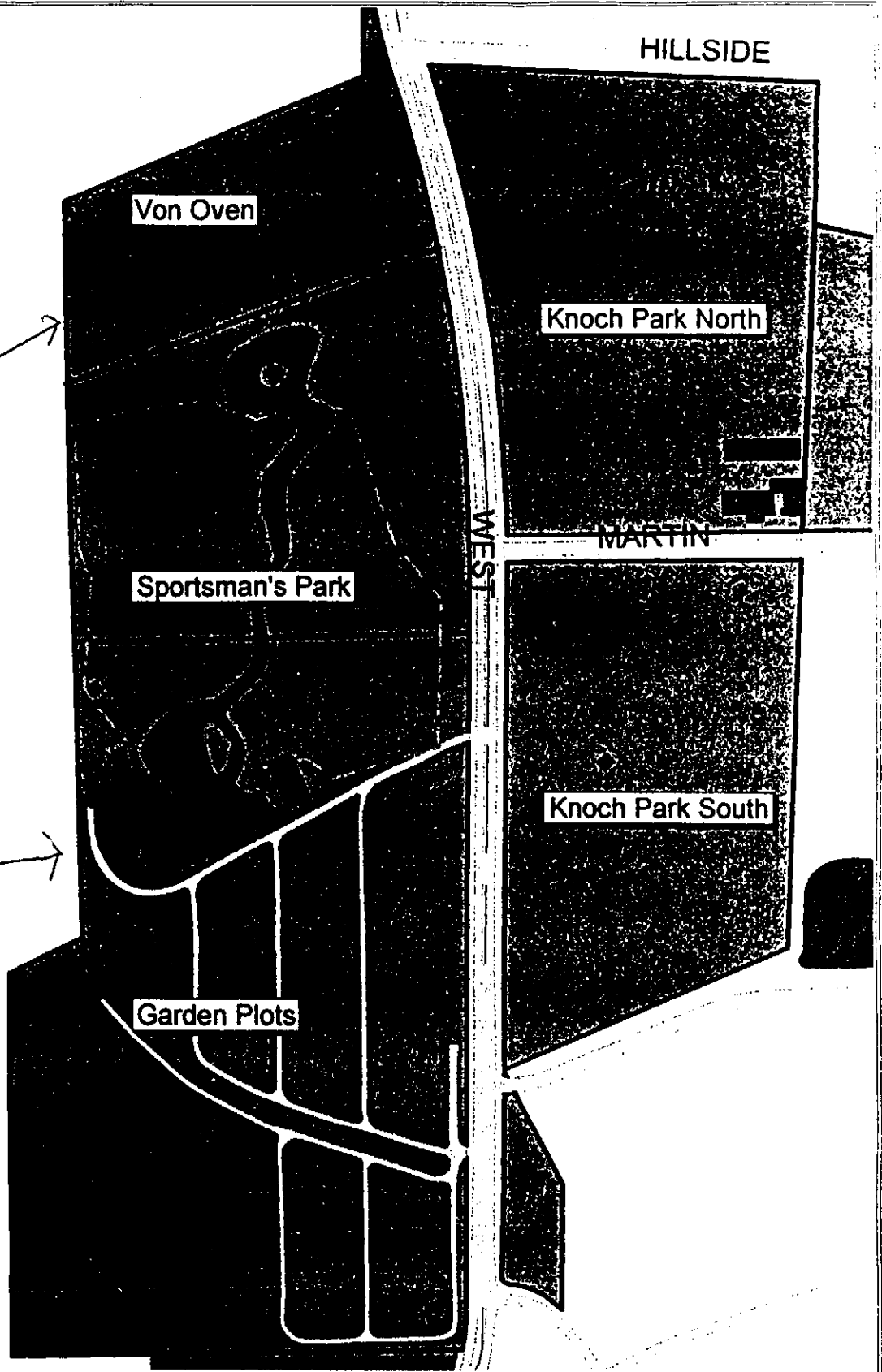




- Park District Structures
- Water
- Sportsman's Fence
- Park Parcel Boundaries

Site Boundary

Site Boundary



Sportsman's Park and Surrounding Area without Aerial Photo

Naperville Park District
Planning Team
August 28, 1998

Scale: 1" = 400'



Appendix B

Example Shaw Field Data Collection Forms

Soil / Sediment Field Logsheet

Site Name:

Project #:

Sample ID:	Sample Location Sketch:
Sample Type*:	
*: SED=Sediment; SUR=Surface soil; SUB=Subsurface Soil; OTH=Other. grab=Grab, comp=Composite	
Date Sampled:	
Time Sampled:	
Depth (ft bgs):	
Physical description:	
Analyses requested:	
	Photograph Log #:
PID:	Calibration Date:
O2/LEL:	Calibration Date:
Weather:	
Temperature:	° F
Sampling Equipment:	
Equipment Decontamination Technique:	
QC Samples:	
Analytical Laboratory:	
Comments:	
Field Technician: (Print)	Date:

Field XRF Screening Data Log Sheet												Sheet _____ of _____
Project Name: <u>NAPERVILLE SPORTSMAN'S PARK</u>												
Project Number: _____												
Date	Reading #	Station I.D.	SampleType	Analyte Pb	1-Sigma Pb +/-	Analyte Cu	1-Sigma Cu +/-	Analyte As	1-Sigma As +/-	Other	1-Sigma	Comment
1												
2												
3												
4												
5												
6												
7												
8												
9												
10												
11												
12												
13												
14												
15												
16												
17												
18												
19												
20												
21												
22												
23												
24												
Additional Field Notes:												
Comment row: i.e. indicate sample character, if sampled in built up area, if bullets/clay targets are visible in sample, etc. Other: if there is a high hit of another analyte....indicate this here												



Shaw[®] Shaw Environmental, Inc.

DAILY FIELD REPORT

[illegible]

CHAIN OF CUSTODY RECORD



**First
Environmental
Laboratories, Inc.**

First Environmental Laboratories

1600 Shore Road, Suite D

Naperville, Illinois 60563

Phone: (630) 778-1200 • Fax: (630) 778-1233

E-mail: firstinfo@firstenv.com

EPA Certification #100292

Project I.D.:

P.O. #:

Matrix Codes: S = Soil W = Water O = Other

[illegible]

FOR LAB USE ONLY:

Cooler Temperature: 0.1-6°C Yes No. _____ °C

Received within 6 hrs. of collection:

Ice Present: Yes___ No___

Sample Refrigerated: Yes ☐ No ☐

Refrigerator Temperature: _____ °C

5035 Vials Frozen: Yes___ No___

Containers Received Preserved: ☐ Yes ☐ NoNeed to meet: IL, TACO ☐ IN, RISC ☐

Notes and Special Instructions:

Relinquished By: _____

Date/Time_

Received By: _____

Date/Time :

Relinquished By: _____

Date/Time_

Received By: _____

Date/Time _

Appendix C

Shaw Standard Operation Procedures (SOP)

STANDARD OPERATING PROCEDURE

Subject: Field Logbook

1. PURPOSE

This procedure is intended to communicate the requirements for selection, use, and maintenance of all field logbooks. Field logbooks are often used to document observations, sampling information, and other pertinent information on project sites. They are considered legal documents and should be maintained and documented accordingly as part of the project file.

2. SCOPE

This procedure is applicable to all Shaw E & I site operations where field logbooks are utilized to document all site activities and pertinent information.

3. REFERENCES

- Nielsen Environmental Field School, 1997, *Field Notebook Guidelines*

4. DEFINITIONS

- **Significant detail**—Any piece and/or pieces of information or an observation that can be considered pertinent to the legal reconstruction of events, description of conditions, or documentation of samples and/or sampling procedures.
- **Significant event**—Any event or events that could influence or be considered pertinent to a specific task or function and therefore require documentation in the Field Logbook.
- **Field Logbook**—Logbooks used at field sites that contain detailed information regarding site activities that must include dates, times, personnel names, activities conducted, equipment used, weather conditions, etc. Field logbooks can be used by a variety of different field personnel and are part of the project file.

5. RESPONSIBILITIES

5.1 Procedure Responsibility

The Field Sampling Discipline Lead is responsible for maintenance, management, and revision of this procedure. Questions, comments, or suggestions regarding this technical SOP should be directed to the Field Sampling Discipline Lead.

5.2 Project Responsibility

Shaw employees performing this task, or any portion thereof, are responsible for meeting the requirements of this procedure. Shaw employees conducting technical review of task performance are also responsible for following appropriate portions of this SOP.

For those projects where the activities of this SOP are conducted, the Project Manager, or designee, is responsible for ensuring that those activities are conducted in accordance with this and other appropriate procedures. Project participants are responsible for documenting information in sufficient

detail to provide objective documentation (i.e. checkprints, calculations, reports, etc.) that the requirements of this SOP have been met. Such documentation shall be retained as project records.

6. PROCEDURE

6.1 General

Each site or operation, as applicable, will have one current Logbook, which will serve as an index of all activities performed at the site or in the task performance. The Logbook is initiated at the start of the first applicable activity. Summary entries are made for every day that covered activities take place. Multiple field logbooks may be used depending upon the number of different types of field personnel conducting work and the various activities at the site. These field logbooks and the site logbooks shall be made part of the project files.

Information recorded in field logbooks includes observations (significant events and details), data, calculations, time, weather, and descriptions of the data collection activity, methods, instruments, and results. Additionally, the field logbook may contain descriptions of wastes, biota, geologic material, and site features including sketches, maps, or drawings as appropriate.

6.2 Equipment and Materials

- Logbook(s), bound with numbered pages, hard-covered, waterproof preferred. One per project or separate significant task (example-treatment residual composite collection).
- Indelible black or dark blue ink pen
- Other items needed to perform required tasks: compass, ruler, calculator, etc.

6.3 Preparation

Site personnel responsible for maintaining field logbooks must be familiar with the SOPs for all tasks to be performed.

Field logbooks are project files and should remain with project documentation when not in use. *Personnel should not keep Field logbooks in their possession when not in use. Field logbooks should only leave the project site for limited periods, and they should always be returned to the site files or the designated on-site location (Sampler's Trailer, etc.).*

Field logbooks shall be bound with lined, consecutively numbered pages. All pages must be numbered prior to initial use of the field logbook.

The front cover shall include the following information:

- Project Number
- Project Name and Task(s) included in logbook
- Dates covered by logbook—the starting date must be entered on the first day of use
- Logbook number—if more than one logbook will be needed to cover project/task(s)

The inside front cover shall contain a listing and sign-off of each person authorized to make entries and/or review the logbook. All persons who make entries or review/approve such entries must signify their authority to enter into the logbook via their signature and the date of their signing on the inside front cover. If initials are used for entries instead of full names, the initials must be entered beside the full name on the inside cover.

6.4 Operation

The following requirements must be met when using a field logbook:

- Record significant details and/or events, work, observations, material quantities, calculations, drawings, and related information directly in the field logbook. If data-collection forms are in use, the information on the form need not be duplicated in the field logbook. However, any forms used to record site information *must be referenced* in the field logbook.
- Information must be factual and unbiased.
- Do not start a new page until the previous one is full or has been marked with a single diagonal line so that additional entries cannot be made. Use both sides of each page.
- Write in black or dark blue indelible ink.
- Do not erase, scribble over, or blot out any entry. Do not use White-Out or like correction items. Before an entry has been signed and dated, changes may be made; however, care must be taken not to obliterate what was written originally. Indicate any deletion by a single line through the material to be deleted. Any change shall be initialed and dated. Error codes (Attachment 1) should be added to the end of the deleted entry. All error codes should be circled.
- Do not remove any pages from the book.
- Do not use loose paper and copy into the field logbook later.
- Record sufficient information to completely document field activities and all significant details/events applicable to the project/task(s) covered by the logbook.
- All entries should be neat and legible.

Specific requirements for field logbook entries include the following:

- Initial and date each page.
- Sign and date the final page of entries for each day.
- Initial, date, and if used, code all changes properly.
- Draw a diagonal line through the remainder of the final page at the end of the day.
- Record the following information on a daily basis:
 - a) Date and time
 - b) Name of individual making entry
 - c) Detailed description of activity being conducted including well, boring, sampling, location number as appropriate
 - d) Unusual site conditions
 - e) Weather conditions (i.e., temperature, cloud cover, precipitation, wind direction and speed) and other pertinent data
 - f) Sample pickup (chain-of-custody form numbers, carrier, time)
 - g) Sampling activities/sample log sheet numbers
 - h) Start and completion of borehole/trench/monitoring well installation or sampling activity

- i) Health and Safety issues, such as PPE upgrades, monitoring results, near-misses, and incidents associated with the logbook areas
- j) Instrumentation calibration details

Entries into the field logbook shall be preceded with the time of the observation. The time should be recorded frequently and at the point of events or measurements that are critical to the activity being logged. All measurements made and samples collected must be recorded unless they are documented by automatic methods (e.g., data logger) or on a separate form required by an operating procedure. In such cases, the field logbook must reference the automatic data record or form.

While sampling, make sure to record observations such as color and odor. Indicate the locations from which samples are being taken, sample identification numbers, the order of filling bottles, sample volumes, and parameters to be analyzed. If field duplicate samples are being collected, note the duplicate pair sample identification numbers. If samples are collected that will be used for matrix spike and/or matrix spike/matrix spike duplicate analysis, record that information in the field logbook.

A sketch of the station location may be warranted. All maps or sketches made in the field logbook should have descriptions of the features shown and a direction indicator. There must be at least one fixed point with measurements on any map drawn. Maps and sketches should be oriented so that north is towards the top of the page.

Other events and observations that should be recorded include (but are not limited to) the following:

- Changes in weather that impact field activities
- Visitors to the site associated with the covered task(s). Note their time of arrival and departure and provide a brief summary of their purpose on site.
- Subcontractor activities applicable to the covered task(s)
- Deviations from procedures outlined in any governing documents, including the reason for the deviation. Deviations from procedures must be accompanied with the proper authorization.
- Significant events that may influence data, such as vehicles in the vicinity of VOC sampling efforts
- Problems, downtime, or delays
- Upgrade or downgrade of personal protective equipment

6.5 Post-Operation

To guard against loss of data due to damage or disappearance of field logbooks, all original completed logbooks shall be securely stored by the project. All field logbooks will be copied at the end of each work shift and attached to the daily reports.

At the conclusion of each activity or phase of site work, the individual responsible for the field logbook will ensure that all entries have been appropriately signed and dated and that corrections were made properly (single lines drawn through incorrect information, initialed, coded, and dated). The completed field logbook shall be submitted to the project records file.

6.6 Restrictions/Limitations

Field logbooks constitute the official record of on-site technical work, investigations, and data collection activities. Their use, control, and ownership are restricted to activities pertaining to specific field operations carried out by Shaw personnel and their subcontractors. They are documents that may be used in court to indicate and defend dates, personnel, procedures, and techniques employed

during site activities. Entries made in these notebooks should be factual, clear, precise, and as non-subjective as possible. Field logbooks, and entries within, are not to be utilized for personal use.

7. ATTACHMENTS

- Attachment 1, Common Data Error Codes

8. FORMS

None.

Attachment 1
Common Data Error Codes

COMMON DATA ERROR CODES

- RE Recording Error
- CE Calculation Error
- TE Transcription Error
- SE Spelling Error
- CL Changed for Clarity
- DC Original Sample Description Changed After Further Evaluation
- WO Write Over
- NI Not Initialed and Dated at Time of Entry
- OB Not Recorded at the Time of Initial Observation

All Error Codes should be circled.

STANDARD OPERATING PROCEDURE

Subject: Field Logsheet

1. PURPOSE

This procedure is intended to communicate the requirements for proper use and completion of Field Logsheets to document sample collection and data gathering activities. Field Logsheets are often utilized to document single location/event information. Examples include boring logs and drum/container logs. This procedure also provides several templates that *may* be utilized or modified to a particular need.

2. SCOPE

This procedure is applicable to all Shaw E & I projects where Field Logsheets are utilized to document data and/or sample collection information. This procedure does **not** mandate the use of Field Logsheets on all Shaw E & I data/sample collection efforts, and projects/programs are free to utilize other means (Field Logbooks, direct data entry, etc.) to document sample collection and other pertinent data gathering activities.

3. REFERENCES

- U.S. Environmental Protection Agency, 1998, *EPA Guidance for Quality Assurance Project Plans*, EPA/600/R-98/018, Washington, D.C.
- U.S. Army Corps of Engineers, 2001, *Requirements for the Preparation of Sampling and Analysis Plans*, EM200-1-3, Washington, D.C.

4. DEFINITIONS

None

5. RESPONSIBILITIES

5.1 Procedure Responsibility

The Field Sampling Discipline Lead is responsible for maintenance, management, and revision of this procedure. Questions, comments, or suggestions regarding this SOP should be directed to the Field Sampling Discipline Lead.

5.2 Project Responsibility

Shaw employees performing this task, or any portion thereof, are responsible for meeting the requirements of this procedure. Shaw employees conducting technical review of task performance are also responsible for following appropriate portions of this SOP.

For those projects where the activities of this SOP are conducted, the Project Manager, or designee, is responsible for ensuring that those activities are conducted in accordance with this and other appropriate procedures. Project participants are responsible for documenting information in sufficient detail to provide objective documentation (i.e. checkprints, calculations,

reports, etc.) that the requirements of this SOP have been met. Such documentation shall be retained as project records.

6. PROCEDURE

Field Logsheets can be prepared to address the specific needs of each project and they can even be converted to laptop data entry forms. Field Logsheets are considered legally defensible, and all appropriate requirements must be observed.

6.1 Required Information

All Field Logsheets must contain entry lines for the following in addition to whatever sample/data gathering-specific information is desired:

- Site/Project Name
- Project Number
- Date (including time if required to properly document)
- Comments or Issues area to record any non-specified information pertinent to the sample/data collection effort
- Initial or signature line for person responsible for completion

6.2 Proper Completion/Use

Whenever Field Logsheets are utilized, the following requirements must be strictly followed and enforced:

- Field Logsheets are to be completed in **real-time**. They should not be filled out by transcription from another source.
- All corrections **must** be single-line cross-out with the initials of the person making the correction.
- All data/information areas **must** be completed. If an entry line/block is not applicable to a particular sample/data gathering effort, this must be indicated on the form by either a single line cross-out or the letters "NA" being written in the data line/block.

7. ATTACHMENTS

None.

8. FORMS

- Container Field Logsheets
- Soil/Sediment Field Logsheets
- Surface Water Field Logsheets
- Air Field Logsheets

Container Field Logsheet
(FS002.1_0)

Date: _____	Time: _____	Site: _____
Container Number: _____	Project #: _____	
Container Size: _____	Weather: _____	
Container Location: _____	Photograph: _____	
Container material of construction: plastic glass metal fiberboard		
Container condition: intact bulging leaking		
Lid type: screw bung ring		
Lid material of construction: plastic glass metal fiberboard		
Labels:	manufacturer: _____	
	address: _____	
	content name: _____	
	chemical name: _____	
	chemical formula: _____	
	other: _____	
Hazard Label:	flammability: _____	
	reactivity: _____	
	health: _____	
	other: _____	
PID: _____		Calibration Date: _____
O2/LEL: _____		Calibration Date: _____
Sampling Device: _____		Decontamination technique: _____
Contents Description:		
	Amount:	1/4 1/2 3/4 full
	Color:	_____
	State:	solid liquid paste other: _____
Sample Number: _____		Preservative: _____
QC Samples: _____		
Analyses requested: _____		
Analytical Laboratory: _____		
Field Technician (Print): _____		
Comments: _____		

Soil/Sediment Field Logsheet
(FS002.2_0)

Site Name:

Project #:

Sample ID:	Sample Location Sketch:
Sample Type*:	
*: SED=Sediment; SUR=Surface soil; SUB=Subsurface Soil; OTH=Other. grab=Grab, comp=Composite	
Date Sampled:	
Time Sampled:	
Depth (ft bgs):	
Physical description:	
Analyses requested:	
	Photograph Log #:
PID:	Calibration Date:
O2/LEL:	Calibration Date:
Weather:	
Temperature: ° F	
Sampling Equipment:	
Equipment Decontamination Technique:	
QC Samples:	
Analytical Laboratory:	
Comments:	
Field Technician: (Print)	Date:

Surface Water Field Logsheet
(FS002.3_0)

Site Name:

Project #:

Sample ID:		Sample Location Sketch:
Date Sampled:		
Time Sampled:		
Depth (ft below surface):		
Analysis	Preservative	
Field Reading	Calibration Date	Photograph Log #: Weather: Temperature: ° F Sampling Equipment: Equipment Decon Technique:
Sp cond:		
pH:		
Temp:		
D.O.:		
Turbidity:		
QC Samples:		
Analytical Laboratory:		
Comments:		
Field Technician: (Print)		Date:

Air Field Logsheet
(FS002.4_0)

Site Name:

Project #:

Sample ID:		Sample Location Sketch:
Date Sampled:		
Time Sampled:		
Sampling Technique:		
Analyses:		
Field Reading	Calibration Date	
		Photograph Log #:
Weather:		
Temperature: ° F		
Sampling Equipment:		
Equipment Decon Technique:		
QC Samples:		
Analytical Laboratory:		
Comments:		
Field Technician: (Print)		Date:

STANDARD OPERATING PROCEDURE

Subject: Chain of Custody Documentation - Paper

1. PURPOSE

The purpose of this procedure is to provide the requirements for completion of written Chain of Custody (COC) documentation and to provide a suggested Chain of Custody Form for project use.

2. SCOPE

This procedure is applicable to all Shaw E & I efforts where samples are transferred among parties, including to off-site testing facilities. Adherence to this procedure is not required whenever the same individual/team is performing the sampling and testing within the same workday, and transfer to the testing process is being documented by other means, e.g. sampling and then field-screening in a mobile laboratory.

3. REFERENCES

- U.S. Environmental Protection Agency, 1986, *Test Methods for Evaluating Solid Waste; Physical/Chemical Methods, SW-846*, Third Edition.
- U.S. Army Corps of Engineers, *Requirements for the Preparation of Sampling and Analysis Plans*, EM200-1-3.
- Shaw E & I, 2002, *Sampler's Training Course Handout*.

4. DEFINITIONS

- **Custody**—The legal term used to define the control and evidence traceability of an environmental sample. A sample is considered to be in an individual's custody when it is in actual physical possession of the person, is in view of the person, is locked in a container controlled by the person, or has been placed into a designated secure area by the person.
- **Chain of Custody Form**—A form used to document and track the custody and transfers of a sample from collection to analysis or placement in a designated secure area within the testing facility.
- **COC Continuation Page**—Additional page(s) that may be included with a Chain of Custody form. The continuation page(s) contain the information on additional samples contained within the *same* cooler/shipping container associated with the cooler/shipping container Chain of Custody form.

5. RESPONSIBILITIES

5.1 Procedure Responsibility

The Field Sampling Discipline Lead is responsible for maintenance, management, and revision of this procedure. Questions, comments, or suggestions regarding this technical SOP should be directed to the Field Sampling Discipline Lead.

5.2 Project Responsibility

Shaw E & I employees performing this task, or any portion thereof, are responsible for meeting the requirements of this procedure. Shaw employees conducting technical review of task performance are also responsible for following appropriate portions of this SOP.

For those projects where the activities of this SOP are conducted, the Project Manager, or designee, is responsible for ensuring that those activities are conducted in accordance with this and other appropriate procedures. Project participants are responsible for documenting information in sufficient detail to provide objective documentation (checkprints, calculations, reports, etc.) that the requirements of this SOP have been met. Such documentation shall be retained as project records.

6. PROCEDURE

6.1 Documentation

All Chain of Custody documentation must be completed in indelible ink. All corrections must be performed using standard single-line cross-out methods, and the initials of the individual making the change must be included beside the corrected entry.

6.2 Continuation Pages

Continuation pages may be utilized for shipping containers/coolers with sufficient samples/sample containers that all of the lines of the Chain of Custody form are used before the documentation of the cooler/shipping container is complete. The number of pages in total must be filled out. *All samples entered onto a Continuation Page must be included in the same cooler/shipping container as those on the Chain of Custody form itself.*

6.3 Header Information

- Each Chain of Custody form must be assigned a unique Reference Document Number—use the Project/proposal number followed by a unique numeric sequence or current date (if only one cooler sent per day). Continuation Pages should contain the same Document Reference Number as the Chain of Custody form that they are associated with. The project team should maintain a log of Chain of Custody Reference Document Numbers.
- The page identifier and total page count section must be completed. Total pages include the Chain of Custody form and any attached Continuation Pages.
- Project number, name, and location information must be completed for all forms.
- If available, the laboratory Purchase Order Number should be included on the appropriate line.
- The name and phone number of the *Project Contact* should be included; the Project Contact should be a responsible individual that the laboratory may access to address analytical issues. This person is usually the analytical lead for the project.
- The *Shipment Date* should be provided on the applicable lines.
- If shipping by carrier, the *Waybill/Airbill Number* must be included. Note: couriers will not sign custody documents. Therefore, inclusion of the waybill/airbill number on the Chain of Custody is the *only* means of documenting the transfer to the carrier.
- Laboratory Destination and Contact information should be provided.

- The Sampler(s) names should be provided on the appropriate line. This line should include all persons whose initials appear on any of the sample containers, to provide the laboratory a means of cross-referencing containers.
- The "Send Report To" information should be completed. If multiple reports/locations are needed, the information should be provided on a separate page included with the Chain of Custody documents.

6.4 Sample Information Section—including on Continuation Page(s)

During actual sampling, each sample must be entered on the COC form at the time of collection in order to document possession. The sampler must not wait until sampling is completed before entering samples on the COC.

- Complete the *Sample ID Number* for each line. If there are multiple container types for a sample, use additional lines to indicate the needed information.
- Ensure that the *Sample Description* matches the description on the sample label—the laboratory will use this information for cross-referencing.
- Provide the *Collection Date* and *Time*. These must match those on the sample label and Field Logbook/Logsheets.
- Indicate whether the sample is a Grab or Composite sample.
- Indicate the *Matrix* of the sample. Use the Matrix Codes listed on the Chain of Custody form.
- Indicate the *Number of Containers* and the *Container Type*. If a sample has multiple container types, use multiple lines and cross-out the information spaces to the left of the container blocks. *Failure to do this may cause the laboratory to log-in each container type as a separate sample/lab-ID, resulting in a confused report and invoice.*
 - Alternatively, if each sample has the same number/type container types, use "various" in the *Container Type* block and provide detail in the *Special Instructions* section, e.g., "Each sample consists of one 16-oz jar, two pre-weighed VOC w/DI water, and one pre-weighed VOC w/Methanol."
- Check the appropriate *Preservative* box for each line/container type.
- Write in and check the *Analyses Requested* boxes for each line/container type. The appropriate method number (e.g., EPA Method 8260C) must be written as well as the method name.
- Indicate the *Turn-around Time Requested* for each sample.
- Use the *Special Instructions* section to provide important information to the laboratory, e.g., samples that may require dilution or samples that will need to be composited by the laboratory. This section may also be used to inform the laboratory of additional information contained in attachments to the Chain of Custody package.
- Circle the appropriate *QC/Data Package Level* requested.

6.5 Custody Transfer Section

- The first *Relinquished By* space must be completed by the individual who will either transfer the samples or seal the shipping container.

- If the samples will be transferred to a courier, write the courier/carrier company in the *Received By* box and enter the Date and Time that the shipping container was closed.
- All other transfers must be performed in person, and the Relinquisher must witness the signing by the Receiver.
- A copy of the Chain of Custody form and all associated Continuation Pages should be maintained in the project files.

7. ATTACHMENTS

None.

8. FORMS

- Shaw E & I Chain of Custody Form
- Shaw E & I COC Continuation Page



Shaw E & I Chain of Custody Form

Ref. Document # _____

Page _____ of _____

--

Project Contact: _____
(Name & phone #)

Send Report To: _____
Phone/Fax Number: _____
Address: _____
City: _____

Project Number: _____
Project Name / Location: _____
Purchase Order #: _____

Shipment Date: _____
Waybill/Airbill Number: _____
Lab Destination: _____
Lab Contact Name / ph. #: _____

Sampler's Name(s): _____

Collection Information

[illegible]

Special Instructions:

QC/Data Package Level Required:

I II III IV/Project Specific: _____

Relinquished By:	Date:
	Time:

Received By:	Date:
	Time:

Relinquished By:	Date:
	Time:

Received By:	Date:
	Time:

Relinquished By:	Date:
	Time:

Received By:	Date:
	Time:

G/C Codes

C = Composite

G = Grab

Matrix Codes

DW = Drinking Water

SO =Soil

GW = Ground Water

SL = Sludge

WW = Waste Water

CP = Chip Samples

SW = Surface Water

WP = Wipe Samples

LIQ = Other Liquid

SOL = Other Solid

AS = Air Sample

SED = Sediment



FS003.1_0

Shaw E & I COC Continuation Page

COC Ref. Document # _____

Page _____ of _____

Project Number: _____

Shipment Date: _____

Project Name / Location: _____

[illegible]

STANDARD OPERATING PROCEDURE

Subject: Custody Seals

1. PURPOSE

The purpose of this procedure is to provide the requirements for completion and attachment of Custody Seals on environmental samples and shipping containers.

2. SCOPE

This procedure is applicable to all Shaw E & I efforts where sample legal defensibility and custody integrity is desired. Adherence to this procedure is not required whenever the same individual/team is performing the sampling and testing within the same workday, and transfer to the testing process is being documented by other means, i.e. sampling and then field-screening in a mobile laboratory.

3. REFERENCES

- U.S. Environmental Protection Agency, 1986, *Test Methods for Evaluating Solid Waste; Physical/Chemical Methods, SW-846*, Third Edition.
- U.S. Army Corps of Engineers, *Requirements for the Preparation of Sampling and Analysis Plans, EM200-1-3*
- Shaw E & I, 2002, Sampler's Training Course Handout.

4. DEFINITIONS

- **Custody**—The legal term used to define the control and evidence traceability of an environmental sample. A sample is considered to be in one's custody if it is in actual physical possession of the person, is in view of the person, has been locked in a container controlled by the person, or has been placed into a designated secure area by the person.
- **Custody Seal**—Commercially available thin strips of adhesive paper with write-in lines for the date/time and identification of the using party. Custody seals are placed over the caps of sample containers and along the cover seals of shipping containers as a means to detect tampering before arrival at the testing facility. All Shaw E & I strategic alliance laboratories provide Custody Seals in their sample container supply kits.

5. RESPONSIBILITIES

5.1 Procedure Responsibility

The Field Sampling Discipline Lead is responsible for maintenance, management, and revision of this procedure. Questions, comments, or suggestions regarding this technical SOP should be directed to the Field Sampling Discipline Lead.

5.2 Project Responsibility

Shaw E & I employees performing this task, or any portion thereof, are responsible for meeting the requirements of this procedure. Shaw E & I employees conducting technical review of task performance are also responsible for following appropriate portions of this SOP.

For those projects where the activities of this SOP are conducted, the Project Manager, or designee, is responsible for ensuring that those activities are conducted in accordance with this and other appropriate procedures. Project participants are responsible for documenting information in sufficient detail to provide objective documentation (i.e. checkprints, calculations, reports, etc.) that the requirements of this SOP have been met. Such documentation shall be retained as project records.

6. PROCEDURE

6.1 Completing the Custody Seal Information

- All Custody Seals must be completed in indelible ink. All corrections must be made using standard single-line cross-out methods, and the initials of the individual making the change must be included beside the corrected entry.
- Each Custody Seal attached must be completed by writing the *Date*, at a minimum, and signing with *full signature* by the person responsible for the sealing of the sample.
- If a space is provided, the *Time* should also be added.

6.2 Attaching the Custody Seals

Whenever possible, custody seals should be attached over the sample container lids during actual sampling and not when the samples are packaged for shipment. This will provide confidence in legal custody and will demonstrate non-tampering during the sample collection process.

Do not attach custody seals to VOC sample containers, as contamination may occur. For these samples, the custody seal should be used to seal the folded plastic zip bag that holds the sample containers.

- For sample jars, the completed Custody Seal should be placed across the top of the lid with the edges below the lid/jar interface and attached to the jar material. This will require the visible breaking of the seal in order to open the container.
- Sample coolers and shipping containers should have Custody Seals attached in such a manner that the seal extends lengthwise from the top edge of the lid to the side of the cooler/container.

7. ATTACHMENTS

None.

8. FORMS

None.

STANDARD OPERATING PROCEDURE

Subject: Sample Labeling

1. PURPOSE

The purpose of this procedure is to provide the requirements for completion and attachment of sample labels on environmental sample containers.

2. SCOPE

This procedure is applicable to all Shaw E & I projects/proposals where samples will be collected.

3. REFERENCES

- U.S. Environmental Protection Agency, 1986, *Test Methods for Evaluating Solid Waste; Physical/Chemical Methods*, SW-846, Third Edition.
- U.S. Army Corps of Engineers, *Requirements for the Preparation of Sampling and Analysis Plans*, EM200-1-3
- Shaw E & I, 2002, Sampler's Training Course Handout.

4. DEFINITIONS

- **Sample Label**—Any writing surface with an adhesive backing that can be used to document sample identification information. The sample label is attached to the sample container as a means of identification and, in some commercially available or laboratory-supplied containers, may be pre-attached. All Shaw E & I strategic alliance laboratories provide sample labels or pre-labeled containers in their sample container supply kits.

5. RESPONSIBILITIES

5.1 Procedure Responsibility

The Field Sampling Discipline Lead is responsible for maintenance, management, and revision of this procedure. Questions, comments, or suggestions regarding this technical SOP should be directed to the Field Sampling Discipline Lead.

5.2 Project Responsibility

Shaw E & I employees performing this task, or any portion thereof, are responsible for meeting the requirements of this procedure. Shaw E & I employees conducting technical review of task performance are also responsible for following appropriate portions of this SOP.

For those projects where the activities of this SOP are conducted, the Project Manager, or designee, is responsible for ensuring that those activities are conducted in accordance with this and other appropriate procedures. Project participants are responsible for documenting information in sufficient detail to provide objective documentation (i.e. checkprints, calculations, reports, etc.) that the requirements of this SOP have been met. Such documentation shall be retained as project records.

6. PROCEDURE

- All sample labels must be completed in indelible ink. All corrections must be performed using standard single-line cross-out methods, and the initials of the individual making the change must be included beside the corrected entry.
- Sample labels should be completed and attached as samples are collected. Do not wait until final packaging to attach and/or complete the sample labels.
- Sample labels must be attached to the non-sealing portion of the container. Do not place labels on or across sample container caps.
- If the laboratory has provided pre-labeled containers, make sure to fill one for each parameter set needed. Laboratory pre-labeled containers are often bar-coded and it is important to provide a complete container set for each sample.
- The following information must be recorded on the Sample Label:
 - Sample Identification Number
 - Date and Time collected
 - Initials of person(s) responsible for collection
- If a space is provided, the *Analysis Requested* should also be added.
- If a *Description* is provided, remember it must match that on the Chain of Custody form for cross-referencing purposes.
- Cover the completed and attached label with clear plastic tape to prevent bleeding of the ink if it becomes wetted. *Do **not** perform this step for pre-weighed VOC vials, as the final weight values will be influenced by the mass of the tape. Protect these containers by enclosing the rack/holder in a plastic bag within the cooler.*

7. ATTACHMENTS

None.

8. FORMS

None.

STANDARD OPERATING PROCEDURE

Subject: Sample Homogenization

1. PURPOSE

The purpose of this procedure is to establish the method for homogenizing samples prior to containerization. Proper homogenization is very important because it helps ensure that sample aliquots are representative of the whole collected sample and helps minimize sampling error so that other errors included in the measurement process, such as laboratory sample preparation and test measurement, can be better assessed.

2. SCOPE

This procedure applies to Shaw Environmental & Infrastructure (Shaw E & I) personnel responsible for the collection of environmental samples. The sample matrix must be amenable to mixing. This SOP applies to the collection of samples that are to be tested for all analytes except volatile analytes.

3. REFERENCES

- American Society for Testing and Materials (ASTM), 1998, Reducing Samples of Aggregate to Testing Size, C702.
- U.S. Army Corps of Engineers, Requirements for the Preparation of Sampling and Analysis Plans, EM 200-1-3, Section E-2, Homogenizing Techniques.

4. DEFINITIONS

- **Homogenize**—The use of physical mixing motions to make a uniform sample matrix.

5. RESPONSIBILITIES

5.1 Procedure Responsibility

The Field Sampling Discipline Lead is responsible for maintenance, management, and revision of this procedure. Questions, comments, or suggestions regarding this technical SOP should be sent to the Field Sampling Discipline Lead.

5.2 Project Responsibility

Shaw employees performing this task, or any portion thereof, are responsible for meeting the requirements of this procedure. Shaw employees conducting technical review of task performance are also responsible for following appropriate portions of this SOP.

For those projects where the activities of this SOP are conducted, the Project Manager, or designee, is responsible for ensuring that those activities are conducted in accordance with this and other appropriate procedures. Project participants are responsible for documenting information in sufficient detail to provide objective documentation (i.e. checkprints, calculations, reports, etc.) that the requirements of this SOP have been met. Such documentation shall be retained as project records.

6. PROCEDURE

Sampling equipment materials shall be selected so as to minimize contamination of samples. Sampling equipment shall be either new (never used previously), documented to have been decontaminated, or dedicated to each specific sampling point. Samples for organic constituent/compound analysis should be collected and mixed using non-reactive material such as glass or stainless steel bowls, trowels, and/or spoons. Samples for metals analysis should be collected and mixed using equipment made of stainless steel, glass, or Teflon®.

Certain types of solid matrices may not be amenable to mixing using conventional techniques. For example, certain solids may require grinding and thorough mixing to ensure that the analytes of interest within the sample are homogeneously distributed. It is extremely important that soil and sediment samples be homogenized to ensure that the entire sample is as representative as possible of the media being sampled.

6.1 Solid Samples

The following two methods are examples for homogenizing solid samples. Other homogenization techniques may be employed using approved standard methods such as ASTM C702, Reducing Samples of Aggregate to Testing Size.

6.1.1 Quartering

- Place the sample on a hard, clean, level surface such as a pan. If such a surface is too small for the desired quantity, a clean sheet of plastic may be used.
- Mix the solid material by turning the entire quantity over three times with a trowel or shovel. For the third time, shovel the material into a cone-shaped pile.
- Carefully press down on the apex of the pile to create a soil layer of uniform thickness and diameter.
- Divide the material in the sample pan or on the plastic into quarters

Option 1

- Mix each quarter individually
- Then mix two quarters to form halves
- Mix each formed half and then fill the appropriate sample jars/containers

Option 2

- Remove two diagonally opposite quarters including any fine material
- Mix the remaining material, build it into a cone, and press down to flatten as before
- Divide the flattened material into quarters, discard two diagonally opposing sections, and repeat
- Repeat the process until only enough sample remains to fill the required containers and proceed to fill the sample jars.

6.1.2 Mixing in a Bowl

- Place the sample in a bowl. Samples for organic constituent/compound analysis should be mixed using bowls and stirrers made of glass or stainless steel, while samples for metals analysis should be mixed using equipment made of glass, stainless steel, or hard plastic. Make sure the bowl is large enough to accommodate the sample, with extra volume to allow for mixing the sample.

- Mix the sample with the stirrer. If round bowls are used for sample mixing, adequate mixing is achieved by stirring the material in a circular fashion, reversing direction, and occasionally turning the material over. High moisture samples are more difficult to homogenize. Use an adequate mixing motion for as long as needed to determine by visual observation that the sample media has taken on a uniform appearance.

6.2 Liquid Samples

Most aqueous samples do not require homogenization since water is well mixed due to diffusion and bulk convection. If the sample matrix is a viscous liquid, semi-solid, or an aqueous one with suspended solids, the sample will require mixing.

Do **not** shake the sample and do not agitate the sample in **any** way if collecting for volatile parameters. Volatile sample containers should be either filled directly from the sample source or if transferring from a large container, such as an automatic sampler reservoir, filled first and **without agitation**.

For non-volatile parameters, mix either using an appropriate stirrer or by gentle swirling and then immediately transfer the material into the appropriate containers. The sample should be mixed frequently during the container-filling step, in particular if there are a large number of containers, so that the condition of the bulk sampled fluid will be approximately the same when each parameter-specific sample container is filled.

7. ATTACHMENTS

None.

8. FORMS

None.

STANDARD OPERATING PROCEDURE

Subject: Compositing

1. PURPOSE

This procedure is intended to provide guidelines for the compositing of samples collected in the course of environmental program activities. Composites represent the average distribution of properties and can be used to reduce analytical costs or represent well-defined decision boundaries.

2. SCOPE

This procedure applies to the compositing of solid and liquid samples where no project-specific process is in place. Field composite methods are not appropriate for Volatile Organic Compounds (VOC) analysis of solids. Composites for these methods must be laboratory derived using either individual grab extracts or other laboratory methods.

3. REFERENCES

- U.S. Environmental Protection Agency, 1987, *Compendium of Superfund Field Operations Methods*, EPA 540/P-87/001a, OSWER 9355.0-14, Washington, DC.
- Shaw E & I Standard Operating Procedure FS010, *Sample Mixing/Homogenization*.

4. DEFINITIONS

- **Composite Sample**—A sample that is comprised of roughly equal amounts of discrete grabs from a set of sample locations or time/flow increments known as a *sample group*.
- **Sample Group**—A predetermined number or time/area span of discrete samples, which is composited into one sample for analytical purposes.

5. RESPONSIBILITIES

5.1 Procedure Responsibility

The Field Sampling Discipline Lead is responsible for maintenance, management, and revision of this procedure. Questions, comments, or suggestions regarding this technical SOP should be sent to the Field Sampling Discipline Lead.

5.2 Project Responsibility

Shaw E & I employees performing this task, or any portion thereof, are responsible for meeting the requirements of this procedure. Shaw E & I employees conducting technical review of task performance are also responsible for following appropriate portions of this SOP.

For those projects where the activities of this SOP are conducted, the Project Manager or designee is responsible for ensuring that those activities are conducted in accordance with this and other appropriate procedures. Project participants are responsible for documenting information in sufficient detail to provide objective documentation (i.e. checkprints, calculations, reports, etc.) that the requirements of this SOP have been met. Such documentation shall be retained as project records.

6. PROCEDURE

The discrete samples that are used to prepare a composite sample must be of equal volume and must each be collected in an identical manner. Field documentation must clearly indicate the composite elements on either a map or a composite logsheet. There are several types of composite samples.

Flow-proportioned composite—Flow-proportioned composite samples are collected proportional to the flow rate during the sampling period by either a time-varying/constant-volume or time-constant/varying-volume method. Flow-proportioned composite samples are typically collected using automatic samplers paced by a flow meter. This sampling method is commonly used for wastewaters.

Time composite—A time composite sample is composed of a discrete number of grab samples collected at equal time intervals during the sampling period. Time composite sampling is often used to sample wastewater discharges or streams.

Volume/mass composite—A volume/mass composite is composed of a discrete number of grab samples collected at defined volume or mass intervals. Volume/mass composite sampling is often used to sample the output of a process system such as a Thermal Destruction Unit or pug mill.

Area composite—Area composite samples are samples collected from individual grab samples located on a regularly spaced grid or along a pile at defined locations and depths. Each of the grab samples must be collected in an identical fashion and must be of equal volume.

Vertical or Depth composite—Vertical composites are composed of individual grab samples collected across a vertical cross section. Like area composites, the grab samples must be collected in an identical fashion and must be of equal volume. Soils and sediments can be used to create vertical composites.

6.1 Solid Composites

- To ensure the integrity of the composite, all discrete grab samples must be collected in an identical manner.
- Composite samples can be created by combining discrete grab samples into the same mixing/holding container as they are collected or by combining and mixing equal aliquots of containerized and homogenized discrete grab samples.
- Remove coarse fragments and organic material from the mixing bowl. Homogenize the sample as specified in SOP FS010, Sample Mixing/Homogenization.
- Remove sample aliquots and place into the appropriate sample containers for shipment to the laboratory.
- Label the sample and document the sampling event according to the project procedures.
- Package/ship the composite sample as required.

6.2 Liquid Composites

- Liquid composite samples should be created by combining equal aliquots of discrete samples.
- Assemble the containers that will comprise a given composite.
- Swirl or stir the individual containers to homogenize the contents just prior to removing the measured aliquots.

- Using clean glass pipets, deliver equal volumes from each grab container to the composite sample container that is to be shipped to the lab. For example, if there are five grab samples, and the composite sample requires 100 mL for the parameter of interest, pipet 20 mL from each of the grab samples into the composite sample container.
- Alternatively, measured volumes can be determined via a graduated cylinder/beaker and combined. The measuring container should be decontaminated between composites.
- Cap/seal the composite container and swirl to agitate. Stirring should be avoided as it increases the risk of introducing contamination to the sample.
- Label the sample(s), document the event, and package/ship the sample(s) as required.

7. ATTACHMENTS

None.

8. FORMS

None.

STANDARD OPERATING PROCEDURE

Subject: Shipping and Packaging of Non Hazardous Samples

1. PURPOSE

The purpose of this procedure is to provide general instructions in the packaging and shipping of non-hazardous samples. The primary use of this procedure is for the transportation of samples collected on site to be sent off site for physical, chemical, and/or radiological analysis.

2. SCOPE

This procedure applies to the shipping and packaging of all non-hazardous samples. Non-hazardous samples are those that do not meet any hazard class definitions found in 49 CFR 107-178, including materials designated as Class 9 materials and materials that represent Reportable Quantities (hazardous substances) and/or materials that are not classified as *Dangerous Goods* under current IATA regulations.

In general most soil, air, and aqueous samples, including those that are acid or caustic preserved do **not** qualify as *hazardous materials* or *dangerous goods*. An exception is methanolic soil VOC vials: these containers are flammable in any quantity and **must** be packaged, shipped, and declared as *Dangerous Goods* whenever transported by air.

The Class 9 "Environmentally Hazardous" designation should only be applied to samples if they are known or suspected (via screening) to contain a sufficient concentration of contaminant to pose a health and/ or environmental risk if spilled in transport. Samples for which screening has shown a potential hazard (i.e. flammability) or those that are derived from a known hazard, including a site/facility with confirmed contamination by an *infectious substance* must also be shipped in accordance with the applicable DOT/IATA requirements. Refer to Shaw E & I SOP FS013.

Improper shipment of hazardous materials, especially willful misrepresentation and shipment as non-hazardous materials, is a violation of federal law and is punishable by fines and possible imprisonment of the guilty parties. It is also a violation of Shaw E & I policy and can result in disciplinary action up to and including termination of employment.

3. REFERENCES

- U.S. Army Corps of Engineers, 2001, *Requirements for the Preparation of Sampling and Analysis Plans*, EM200-1-3, Washington, D.C.
- U.S. Department of Transportation Regulations, 49 CFR Parts 108-178
- International Air Transport Association (IATA), *Dangerous Goods Regulations*, current edition.

4. DEFINITIONS

- **Cooler/Shipping Container**—Any hard-sided insulated container meeting DOT's or IATA's general packaging requirements.
- **Bubble Wrap**—Plastic sheeting with entrained air bubbles for protective packaging purposes.

5. RESPONSIBILITIES

5.1 Procedure Responsibility

The Field Sampling Discipline Lead is responsible for maintenance, management, and revision of this procedure. Questions, comments, or suggestions regarding this technical SOP should be sent to the Field Sampling Discipline Lead.

5.2 Project Responsibility

Shaw employees performing this task, or any portion thereof, are responsible for meeting the requirements of this procedure. Shaw employees conducting technical review of task performance are also responsible for following appropriate portions of this SOP.

For those projects where the activities of this SOP are conducted, the Project Manager, or designee, is responsible for ensuring that those activities are conducted in accordance with this and other appropriate procedures. Project participants are responsible for documenting information in sufficient detail to provide objective documentation (i.e. checkprints, calculations, reports, etc.) that the requirements of this SOP have been met. Such documentation shall be retained as project records.

6. PROCEDURE

6.1 Packaging

- Use tape and seal off the cooler drain on the inside and outside to prevent leakage.
- Place packing material on the bottom on the shipping container (cooler) to provide a soft impact surface.
- Place a large (30-55 gallon or equivalent) plastic bag into the cooler (to minimize possibility of leakage during transit).
- Starting with the largest glass containers, wrap each container with sufficient bubble wrap to ensure the best chance to prevent breakage of the container.
- Pack the largest glass containers in the bottom of the cooler, placing packing material between each of the containers to avoid breakage from bumping.
- Double-bag the ice (chips or cubes) in gallon- or quart-sized resealable plastic freezer bags and wedge the ice bags between the sample bottles.
- Add bagged ice across the top of the samples.
- When sufficiently full, seal the inner protective plastic bag, and place additional packing material on top of the bag to minimize shifting of containers during shipment.
- Tape a gallon-sized resealable plastic bag to the inside of the cooler lid, place the completed chain of custody document inside, and seal the bag shut.
- Tape the shipping container (cooler) shut using packing tape, duct tape, or other tear-resistant adhesive strips. Taping should be performed to ensure the lid cannot open during transport.
- Place a custody seal on two separate portions of the cooler, to provide evidence that the lid has not been opened prior to receipt by the intended recipient.

6.2 Labeling

- A “This Side Up” arrow should be adhered to all sides of the cooler, especially ones without obvious handles.
- The name and address of the receiver and the shipper must be on the top of the cooler.
- The airbill must be attached to the top of the cooler.

6.3 Shipping Documentation

- A Cooler Shipment Checklist (Attachment 1) should be completed and kept in the project file.


7. ATTACHMENTS

- Attachment 1, Shaw E & I Cooler Shipment Checklist

8. FORMS

None.

**Attachment 1
Shaw E & I Cooler Shipment Checklist**

 Shaw TM Shaw E & I, Inc.	Project Name _____	Project Number _____
	Address _____	Date _____ Time _____
	City, State, Zip _____	Fax No. _____
	Site Contact No. _____	

SAMPLE CHECKLIST

	YES	NO	COMMENTS
SAMPLE LIDS ARE TIGHT AND CUSTODY SEALS IN PLACE?	<input type="checkbox"/>	<input type="checkbox"/>	_____
ARE ALL SAMPLE NUMBERS, DATES, TIMES AND OTHER LABEL INFORMATION LEGIBLE AND COMPLETE?	<input type="checkbox"/>	<input type="checkbox"/>	_____
HAVE ALL SAMPLE NUMBERS, DATES, TIMES AND OTHER SAMPLING DATA BEEN LOGGED INTO THE SAMPLE LOG BOOK?	<input type="checkbox"/>	<input type="checkbox"/>	_____
DO SAMPLE NUMBERS AND SAMPLE DESCRIPTIONS ON THE LABELS MATCH THOSE ON THE COC?	<input type="checkbox"/>	<input type="checkbox"/>	_____
HAVE THE SAMPLES BEEN PROPERLY PRESERVED?	<input type="checkbox"/>	<input type="checkbox"/>	_____
HAVE THE CHAIN OF CUSTODIES BEEN FILLED OUT COMPLETELY AND CORRECTLY?	<input type="checkbox"/>	<input type="checkbox"/>	_____
DOES THE ANALYTICAL SPECIFIED ON THE COC MATCH THE ANALYTICAL SPECIFIED IN THE SCOPE OF WORK?	<input type="checkbox"/>	<input type="checkbox"/>	_____
HAVE THE COC'S BEEN PROPERLY SIGNED IN THE TRANSFER SECTION?	<input type="checkbox"/>	<input type="checkbox"/>	_____

PACKAGING CHECKLIST

	YES	NO	COMMENTS
HAS EACH SAMPLE BEEN PLACED INTO AN INDIVIDUAL PLASTIC BAG?	<input type="checkbox"/>	<input type="checkbox"/>	_____
HAS THE DRAIN PLUG OF THE COOLER BEEN TAPED CLOSED WITH WATER PROFF TAPE FROM THE INSIDE?	<input type="checkbox"/>	<input type="checkbox"/>	_____
HAVE ALL THE SAMPLES BEEN PLACED INTO THE COOLER IN AN UPRIGHT POSITION?	<input type="checkbox"/>	<input type="checkbox"/>	_____
IS THERE ADEQUATE SPACING OF SAMPLES SO THAT THEY WILL NOT TOUCH DURING SHIPMENT?	<input type="checkbox"/>	<input type="checkbox"/>	_____
HAVE AN ADEQUATE NUMBER OF BLUE ICE PACKS OR WATER ICE BEEN PLACED AROUND AND ON TOP OF THE SAMPLE?	<input type="checkbox"/>	<input type="checkbox"/>	_____
HAS FRESH BLUE ICE OR WATER ICE BEEN ADDED TO THE COOLER THE DAY OF THE SHIPMENT?	<input type="checkbox"/>	<input type="checkbox"/>	_____
HAS THE COOLER BEEN FILLED WITH ADDITIONAL CUSHIONING MATERIAL?	<input type="checkbox"/>	<input type="checkbox"/>	_____
HAS THE COC BEEN PLACE IN A ZIPLOCK BAG AND TAPED TO THE INSIDE OF THE LID OF THE COOLER?	<input type="checkbox"/>	<input type="checkbox"/>	_____
HAVE CUSTODY SEALS BEEN PLACED ONTO THE LID?	<input type="checkbox"/>	<input type="checkbox"/>	_____
HAS THE COOLER BEEN LABELED "THIS SIDE UP"?	<input type="checkbox"/>	<input type="checkbox"/>	_____
IF REQUIRED, HAS THE COOLER BEEN LABELED WITH THE DOT PROPER SHIPPING NAME, UN NUMBER AND LABEL?	<input type="checkbox"/>	<input type="checkbox"/>	_____
HAS THE LABORATORY PERFORMING THE ANALYSES BEEN NOTIFIED OF THE SHIPMENT OF SAMPLES?	<input type="checkbox"/>	<input type="checkbox"/>	_____

PROBLEMS/RESOLUTIONS: _____

PREPARED BY: _____ SIGNATURE _____

STANDARD OPERATING PROCEDURE

Subject: Packaging and Shipping of DOT/IATA-Hazardous Samples

1. PURPOSE

The purpose of this procedure is to provide general instructions for packaging and shipping of hazardous samples, as defined by DOT and/or IATA, including Class 9 “Environmentally hazardous substances.” The primary use of this procedure is for the transportation of samples collected on site to be sent off site for physical, chemical, biological (*infectious substance*), and/or radiological analysis in accordance with applicable laws and regulations and without destroying sample integrity.

2. SCOPE

This procedure applies to the packaging and shipping of all DOT/IATA-hazardous samples. Samples must be packaged and shipped as hazardous materials if they meet any of the hazard class definitions in 49 CFR 107-178, including Reportable Quantities, and/or if they can be classified as a *Dangerous Good* under IATA. All IATA classified materials designated for air transport, even in Limited Quantities, **must** be declared, packaged, and shipped as *Dangerous Goods*. Examples include methanolic VOC soil samples and any samples from a project/facility known to be impacted by an *infectious substance*.

Improper shipment of hazardous materials, especially willful misrepresentation and shipment as non-hazardous materials, is a violation of federal law and is punishable by fines and possible imprisonment of guilty parties. It is also a violation of Shaw E & I policy and can result in disciplinary action up to and including termination of employment.

3. REFERENCES

- U.S. Army Corps of Engineers, 2001, *Requirements for the Preparation of Sampling and Analysis Plans*, EM200-1-3, Washington, D.C.
- U.S. Department of Transportation Regulations, 49 CFR Part 107-178
- *Dangerous Goods Regulations*, current edition, International Air Transport Association (IATA)

4. DEFINITIONS

- **Dangerous Goods Airbill**—Form required when offering *Dangerous Goods* as defined in IATA regulations for air transport. The “*Dangerous Goods Airbill*” must be completed and signed by a responsible and qualified person. Some carriers require a typed or computer-generated form.
- **Inner packaging**—Packaging in immediate contact with the hazardous materials to be shipped, such as a sample jar or vial.
- **Limited Quantity**—In the IATA Tables, the maximum total amount of a *Dangerous Good* that can be transported without using UN-specification containers, such as a non-UN tested cooler.

- **Outer packaging**—Packaging into which one or more inner packages can be placed, such as a sturdy plastic cooler meeting general packaging requirements or a 5-gallon UN-specification plastic pail.
- **Performance-Oriented Packaging**—Packaging designed for and tested to be used for shipment of DOT-hazardous materials. Also known as “UN-specification” packaging.
- **Qualified person**—An individual with appropriate DOT/IATA Hazardous Materials training, including General Awareness, Function-Specific, and Safety training, necessary to properly classify samples as hazardous materials and to complete all subsequent shipping steps.

5. RESPONSIBILITIES

5.1 Procedure Responsibility

The Field Sampling Discipline Lead is responsible for the maintenance, management, and revision of this procedure. Questions, comments, or suggestions regarding this technical SOP should be directed to the Field Sampling Discipline Lead.

5.2 Project Responsibility

Shaw E & I employees performing this task, or any portion thereof, are responsible for meeting the requirements of this procedure. Shaw E & I employees conducting technical review of task performance are also responsible for following appropriate portions of this SOP.

For those projects where the activities of this SOP are conducted, the Project Manager, or designee, is responsible for ensuring that those activities are conducted in accordance with this and other appropriate procedures. Project participants are responsible for recording information in sufficient detail to provide objective documentation (i.e. checkprints, calculations, reports, etc.) that the requirements of this SOP have been met. Such documentation shall be retained as project records.

6. PROCEDURE

A Qualified Person **must** perform or oversee the classification, packaging, and completion of all related declaration and shipping papers. *It is a violation of federal law to pre-complete these documents and provide them to an unqualified person without providing minimal training to that person.* This training **must** be documented and may take the form of a verbal discussion, hands-on demonstration, or detailed written instructions, including a task-specific SOP, with review provided by the Qualified Person.

The basic packaging and shipping procedures are as follows:

- Determine the traits of the material to be shipped and compare them to the specific hazard class definitions in the appropriate regulations. If the material falls within one or more hazard class definitions, it is deemed “hazardous”. Select the most accurate proper shipping name and packing group combination, and prepare the package according to the prescribed requirements for quantity limitations, authorized packaging, marking, labeling, and documentation.
- Check the current IATA regulations to make sure the carrier accepts the material(s) and/or does not have its own special requirements for shipment.
- If shipping multiple inner packages that each meet a separate hazard class definition, consult the “Separation and Segregation” table in the appropriate regulations for guidance on

packaging and prepare as an over-pack with individual marking and labeling on the outer packaging.

- If shipping multiple inner packages that meet the same hazard class(es) but represent both solid and liquid matrices, prepare as an over-pack with individual marking and labeling on the outer packaging.
- If shipping hazardous material that meets more than one hazard class definition, check the hazard precedence table in the appropriate regulations to determine primary and subsidiary classes.

6.1 Additional Inner Packaging Requirements

- Place each sample container into a resealable plastic baggie.
- Fold over and tape the bag seal onto the sample jar to prevent the closure from unsealing.
- Several IATA packing instructions require containerizing of glass/plastic sample jars into a sealed primary receptacle such as a metal can before placing them into outer-packaging, i.e. the cooler.
 - Wrap the bagged sample container with bubble-wrap or other packing material to prevent breakage against the sides of the primary receptacle, and place it into the primary receptacle.
 - Seal the primary receptacle and label it with the Sample ID and any hazard information and place it into a plastic bag to protect the label.

6.2 Additional Outer Packaging Requirements

- Samples that in total qualify as Excepted Quantities or Limited Quantities do not require the use of UN-specification packaging and may be shipped in sturdy coolers, pails, or any packaging that meets general packaging requirements.
- Samples that do not qualify as Excepted Quantities or Limited Quantities require UN-specification packaging. For such samples that also require cooling to meet sample preservation requirements, UN-specification coolers are available from several Haz-Mat packaging vendors.
- If using a cooler of any kind, seal off the cooler drain on the inside and outside with tape to prevent leakage.
- Place cushioning and/or absorbent material on the bottom of the outer packaging to provide a soft impact surface.
- Place a plastic bag into the container (to minimize the possibility of leakage during transit).
- Wrap glass inner packagings with sufficient bubble wrap to ensure the best chance to prevent breakage of the container.
- For methanolic soil VOC vials, place vials into the supplied rack/holder or box and then place it into a tied off plastic bag to keep out moisture.
- Pack the largest inner packagings in the bottom of the container with cushioning material between each to avoid breakage from bumping.
- If cooling is required, double-bag the ice (chips or cubes) in gallon- or quart-size freezer Ziploc-type resealable plastic bags, and wedge the ice bags between the inner packages and/or primary receptacles. Also add bagged ice across the top of the samples/receptacles.

- When sufficiently full, seal the plastic bag that lines the outer packaging, and place additional cushioning material on top of the bag to minimize shifting of contents during shipment.
- Tape a gallon Ziploc-type bag to the inside of the container lid, place the completed chain of custody document inside, and seal the bag shut.
- Tape the outer packaging closed using packing tape, duct tape, or other tear-resistant adhesive strips.
- Place a custody seal on two separate portions of the outer packaging to provide evidence that the lid remains sealed during transit.

6.3 Marking and Labeling

- If the package contains any liquids, orientation arrows must be applied to two opposite faces of the package (front and back or both ends).
- The proper shipping name, UN number, and all other required markings, as well as the appropriate hazard class label, must be placed on the same face of the package in close proximity to each other.
- Consignor and consignee information should appear on some face of the package in addition to appearing on the shipping papers that are enclosed in a pouch attached to the package.

6.4 Shipping Documentation

- If a sturdy cooler is used, whether UN-specification or not, complete a Cooler Shipment Checklist (see Attachment 1) and keep it in the project file.
- A Dangerous Goods Airbill must be completed, inserted into an adhesive pouch, and attached to the package in close proximity to the proper shipping name and hazard class label.
- Many carriers require a typed or computer-generated Dangerous Goods Airbill.
- If the Dangerous Goods Airbill has an area specifically designated for a "24-Hour Emergency Response" telephone number, insert "800-424-9300" into that space. If it does not, write "24-Hour Emergency Response Telephone Number: 800-424-9300" in the "Additional Handling Information" section of the airbill. Immediately following the telephone number, write "ERG-xxx," where xxx is the 3-digit Emergency Response Guidebook page number that corresponds to the hazardous material being shipped.
- The shipper must sign the certification on the airbill.
- Prior to carrier pickup, a copy of the Dangerous Goods Airbill must be faxed to CHEMTREC at 703-741-6037 with a Shaw coversheet addressed to "ITCR."


7. ATTACHMENTS

- Attachment 1, Shaw E & I Cooler Shipment Checklist

8. FORMS

None.

**Attachment 1
Shaw E & I Cooler Shipment Checklist**

 Shaw E & I, Inc.	Project Name _____	Project Number _____
	Address _____	Date _____ Time _____
	City, State, Zip _____	Fax No. _____
	Site Contact No. _____	

SAMPLE CHECKLIST	YES	NO	COMMENTS
SAMPLE LIDS ARE TIGHT AND CUSTODY SEALS IN PLACE?	<input type="checkbox"/>	<input type="checkbox"/>	_____
ARE ALL SAMPLE NUMBERS, DATES, TIMES AND OTHER LABEL INFORMATION LEGIBLE AND COMPLETE?	<input type="checkbox"/>	<input type="checkbox"/>	_____
HAVE ALL SAMPLE NUMBERS, DATES, TIMES AND OTHER SAMPLING DATA BEEN LOGGED INTO THE SAMPLE LOG BOOK?	<input type="checkbox"/>	<input type="checkbox"/>	_____
DO SAMPLE NUMBERS AND SAMPLE DESCRIPTIONS ON THE LABELS MATCH THOSE ON THE COC?	<input type="checkbox"/>	<input type="checkbox"/>	_____
HAVE THE SAMPLES BEEN PROPERLY PRESERVED?	<input type="checkbox"/>	<input type="checkbox"/>	_____
HAVE THE CHAIN OF CUSTODIES BEEN FILLED OUT COMPLETELY AND CORRECTLY?	<input type="checkbox"/>	<input type="checkbox"/>	_____
DOES THE ANALYTICAL SPECIFIED ON THE COC MATCH THE ANALYTICAL SPECIFIED IN THE SCOPE OF WORK?	<input type="checkbox"/>	<input type="checkbox"/>	_____
HAVE THE COC'S BEEN PROPERLY SIGNED IN THE TRANSFER SECTION?	<input type="checkbox"/>	<input type="checkbox"/>	_____

PACKAGING CHECKLIST	YES	NO	COMMENTS
HAS EACH SAMPLE BEEN PLACED INTO AN INDIVIDUAL PLASTIC BAG?	<input type="checkbox"/>	<input type="checkbox"/>	_____
HAS THE DRAIN PLUG OF THE COOLER BEEN TAPED CLOSED WITH WATER PROFF TAPE FROM THE INSIDE?	<input type="checkbox"/>	<input type="checkbox"/>	_____
HAVE ALL THE SAMPLES BEEN PLACED INTO THE COOLER IN AN UPRIGHT POSITION?	<input type="checkbox"/>	<input type="checkbox"/>	_____
IS THERE ADEQUATE SPACING OF SAMPLES SO THAT THEY WILL NOT TOUCH DURING SHIPMENT?	<input type="checkbox"/>	<input type="checkbox"/>	_____
HAVE AN ADEQUATE NUMBER OF BLUE ICE PACKS OR WATER ICE BEEN PLACED AROUND AND ON TOP OF THE SAMPLE?	<input type="checkbox"/>	<input type="checkbox"/>	_____
HAS FRESH BLUE ICE OR WATER ICE BEEN ADDED TO THE COOLER THE DAY OF THE SHIPMENT?	<input type="checkbox"/>	<input type="checkbox"/>	_____
HAS THE COOLER BEEN FILLED WITH ADDITIONAL CUSHIONING MATERIAL?	<input type="checkbox"/>	<input type="checkbox"/>	_____
HAS THE COC BEEN PLACE IN A ZIPLOCK BAG AND TAPED TO THE INSIDE OF THE LID OF THE COOLER?	<input type="checkbox"/>	<input type="checkbox"/>	_____
HAVE CUSTODY SEALS BEEN PLACED ONTO THE LID?	<input type="checkbox"/>	<input type="checkbox"/>	_____
HAS THE COOLER BEEN LABELED "THIS SIDE UP"?	<input type="checkbox"/>	<input type="checkbox"/>	_____
IF REQUIRED, HAS THE COOLER BEEN LABELED WITH THE DOT PROPER SHIPPING NAME, UN NUMBER AND LABEL?	<input type="checkbox"/>	<input type="checkbox"/>	_____
HAS THE LABORATORY PERFORMING THE ANALYSES BEEN NOTIFIED OF THE SHIPMENT OF SAMPLES?	<input type="checkbox"/>	<input type="checkbox"/>	_____

PROBLEMS/RESOLUTIONS: _____

PREPARED BY: _____ SIGNATURE _____

STANDARD OPERATING PROCEDURE

Subject: Decontamination of Contact Sampling Equipment

1. PURPOSE

This procedure is intended to provide minimal guidelines for the decontamination of contact sampling equipment. Contact sampling equipment is equipment that comes in direct contact with the sample or the portion of a sample that will undergo chemical analyses or physical testing.

2. SCOPE

This procedure applies to all instances where non-disposable direct contact sampling equipment is utilized for sample collection and no project-specific procedure is in place. This procedure is not intended to address decontamination of peristaltic or other sampling pumps and tubing. The steps outlined in this procedure must be executed between each distinct sample data point.

3. REFERENCES

- U.S. Environmental Protection Agency, Region 4, 2001, *Environmental Investigations Standard Operating Procedures and Quality Assurance Manual*, 980 College Station Road, Athens, Georgia. November.
- US Army Corp of Engineers, Washington, D.C., 2001, Requirements for the Preparation of Sampling and Analysis Plans (EM-200-1-3), February.

4. DEFINITIONS

- **Soap**—A standard brand of phosphate-free laboratory detergent, such as Liquinox®.
- **Organic Desorbing Agent**—A solvent used for removing organic compounds. The specific solvent would depend upon the type of organic compound to be removed. See Attachment 1 for recommendations.
- **Inorganic Desorbing Agent**—An acid solution for use in removing trace metal compounds. The specific acid solution would depend upon the type of inorganic compound to be removed. See Attachment 1 for recommendations.
- **Tap water**—Water obtained from any municipal water treatment system. An untreated potable water supply can be used as a substitute for tap water if the water does not contain the constituents of concern.
- **Distilled Water**—Water that has been purified via distillation. Distilled water can be purchased in most stores and is acceptable as a final rinse in non-trace analytical decontamination processes. Examples would include disposal profiling, HazCat, and other gross screening applications.
- **Analyte-free water**—Water that has been treated by passing through a standard deionizing resin column, and for organics either distillation or activated carbon units. At a minimum, the finished water should contain no detectable heavy metals or other inorganic compounds, and/or no detectable organic compounds (i.e., at or above analytical detection limits). Type I and Type II Reagent Grade Water meet this definition as does most laboratory-supplied blank water.

5. RESPONSIBILITIES

5.1 Procedure Responsibility

The Field Sampling Discipline Lead is responsible for maintenance, management, and revision of this procedure. Questions, comments, or suggestions regarding this technical SOP should be sent to the Field Sampling Discipline Lead.

5.2 Project Responsibility

Shaw employees performing this task, or any portion thereof, are responsible for meeting the requirements of this procedure. Shaw employees conducting technical review of task performance are also responsible for following appropriate portions of this SOP.

For those projects where the activities of this SOP are conducted, the Project Manager, or designee, is responsible for ensuring that those activities are conducted in accordance with this and other appropriate procedures. Project participants are responsible for documenting information in sufficient detail to provide objective documentation (checkprints, calculations, reports, etc.) that the requirements of this SOP have been met. Such documentation shall be retained as project records.

6. PROCEDURE

Wear appropriate eye protection including safety goggles when working with corrosive liquids, especially when diluting concentrated materials to create low-percentage solutions and follow all project Health and Safety requirements. Decontamination wastes are to be recovered and handled as impacted project waste materials and must be disposed of in accordance with regulatory requirements.

A decontamination area should be established. Implements can either be immersed in a 5-gallon bucket containing each solution/rinse or the solutions can be contained in hand-held units made of an inert and compatible material; such as a Teflon™ wash bottle. The analyte-free water needs to be placed in a container that will be free of any compounds of concern.

Consult Attachment 1 for the decontamination solutions/solvents appropriate to the task. The minimum steps for decontamination are as follows:

1. Remove particulate matter and other surface debris by brushing and/or dipping in the soap solution.
2. Rinse thoroughly with tap water.
3. If necessary, rinse with other applicable solutions/solvents. If hexane is used, be sure to follow it with isopropyl alcohol to allow for the final water rinses to properly mix and contact the surface.
4. Final rinse three times to make sure all residual solutions/solvents are removed.
5. Place decontaminated equipment on a clean surface appropriate for the compounds of concern and allow to air dry.

7. ATTACHMENTS

- Attachment 1, Recommended Decontamination Procedures.

8. FORMS

None.

Attachment 1
Recommended Decontamination Procedures

Compound	Detergent Wash	Tap Water	Inorganic Desorbing Agent	Tap Water	Organic Desorbing Agent ¹	Final Water Rinse ⁴	Air Dry
Organic Constituents							
Volatile Organic Compounds	✓	✓			Methanol Purge & Trap grade	✓	✓
Base Neutrals/Acid Extractables/PCBs/Pesticides	✓	✓			Hexane followed by Isopropyl Alcohol	✓	✓
Organic Bases ²	✓	✓	1% nitric acid	✓	Isopropyl Alcohol	✓	✓
Organic Acids ³	✓	✓	1% nitric acid		Isopropyl Alcohol	✓	✓
Inorganic Constituents							
Trace Metals and Radio Isotopes	✓	✓	10% Nitric acid -Trace metals grade	✓		✓	✓
Cations/Anions	✓	✓				✓	✓
Acidic Compounds	✓	✓				✓	✓
Basic Compounds (caustic)	✓	✓	1% nitric acid	✓		✓	✓

1 – All organic solvents must be Pesticide Grade or better. The selection of appropriate solvent rinses should first consider if a *known or suspected* contaminant requires removal from sampling equipment. Secondly, identify whether the subsequent analytical protocol would be impacted by the proposed solvent or an impurity thereof (e.g., residual acetone present in isopropyl alcohol would be measured with certain volatile organics analysis).

2 - Organic bases include amines, hydrazines.

3 - Organic acids include phenols, thiols, nitro and sulfonic compounds.

4- Use a grade of water appropriate to the application. For trace level analysis this must be Analyte Free Water. For non-trace applications store-bought distilled water is sufficient

Adapted from: Appendix E, Requirements for the Preparation of Sampling and Analysis Plans (EM-200-1-3), February 2001. US Army Corp of Engineers, Washington, D.C.

Revision 1- 3/2006

STANDARD OPERATING PROCEDURE

Subject: Data Usability Review

1. PURPOSE

The purpose of this procedure is to establish the means by which all subcontracted environmental analytical data will be reviewed for completeness and usability based upon comparison to the project action/decision levels and Data Quality Objectives before use in the intended decision-making processes.

2. SCOPE

This procedure applies to all subcontracted analytical data including faxed or e-mailed preliminary reports.

By way of its requirements, this procedure prohibits verbal communication of analytical results and establishes minimum deliverable standards that must be provided for all subcontracted analytical data reports—including faxed or e-mailed preliminary reports. These minimum standards include the following:

- Sample Results
- Chain of Custody – unless already available to the reviewer
- Sample Receipt Documentation – unless already available to the reviewer
- QC Summary – Laboratory Control Blank, Laboratory Control Spike, Matrix Spike, Matrix Spike Duplicate, Post-digest Spike
- Surrogate Summary – (if applicable)
- Hold-time Compliance Summary – or signed certification that all requirements were met
- Initial and Continuing Calibration Information – or signed certification that it meets prescribed requirements
- GC/MS Tuning Information – (if applicable) or signed certification that it meets prescribed requirements

This procedure should be performed only by or under the oversight of properly qualified individuals. Oversight may be accomplished through provision of a project-specific and well-defined checklist, training in its use, regular QA checks, and real-time availability for issue resolution.

3. REFERENCES

- U.S. Environmental Protection Agency, *National Functional Guidelines for Inorganic Data Review*, EPA 540/R-94-013.
- U.S. Environmental Protection Agency, *National Functional Guidelines for Organic Data Review*, EPA 540/R-94-012.

- U.S. Department of Defense, 2002, Department of Defense Quality Systems Manual for Environmental Laboratories, Final, June.
- U.S. Army Corps of Engineers, Requirements for the Preparation of Sampling and Analysis Plans, EM-200-1-3.

4. DEFINITIONS

- **Data Usability Review (DUR)**—The cursory review of an analytical data package for completeness and compliance with the ordered analysis, specified quality, and method/project-specific protocols before the data is used as input to a particular project decision-making process. The DUR process identifies any potential data quality issues and informs the data users of the effect on the data usability.
- **Data Quality Objectives**—The empirical statements and quantitative measures necessary for a given set of measurements to be usable in the planned decision.
- **Data Quality Indicators**—Field and laboratory measures for which compliance with specified requirements or limits can be construed to support attainment of the Data Quality Objectives in a given data set.
- **Analytical Data Package**—The manner in which analytical results are provided from subcontractor laboratories. Analytical Data Packages can be received via fax, e-mail, or postal mail.
- **QC Summary**—A summary table of laboratory QC sample results.
- **Laboratory Control Blank (LCB)**—Reagent Water or Clean Solid Matrix analyzed in the same manner as a sample to determine the Target Analyte concentration contribution due to contamination in the entire analytical system.
- **Laboratory Control Spike (LCS)**—Reagent Water or Clean Solid Matrix spiked with a known concentration of target analytes and analyzed as a sample to determine the method accuracy of the analytical system.
- **Matrix Spike**—A sample spiked with a known concentration of target analyte and analyzed along with the rest of the analytical batch. The percent recovery of the target analytes is used to determine the effect on accuracy due to the sample matrix.
- **Matrix Spike Duplicate**—A duplicate of the Matrix Spike used to determine the analytical precision, expressed as Relative Percent Difference (RPD) of the analytical system.
- **Surrogate Compound**—In several organic methods, a compound similar in structure and chemical behavior to the target analytes, which is added to each Sample and QC Sample at a known concentration before the analysis begins. The surrogate recovery is used to approximate the recovery of the target compounds based upon the behavior of chemically similar analytes.
- **Post-digest Spike**—In metals analyses, used to determine the possibility of chemical interferences and digestion deficiencies. If the normal QC results are unacceptable, a known concentration of the target analyte is added to the sample digestate. The recovery is then used to determine if reanalysis or data qualification is warranted.
- **QC Acceptance Range**—The limits that define QC results demonstrating compliant accuracy and precision.
- **Qualified Person**—An individual capable through knowledge, education, formal training, and/or experience in the establishment and verification of analytical Data Quality Objectives. The

Qualified Person is usually a chemist or environmental professional with several years of environmental analytical experience.

- **Trip Blank**—In VOC analysis, a container of Reagent Grade Water that is included in the sample cooler and analyzed by the laboratory to determine if cross-contamination may have occurred in shipping.
- **Ambient or Field Blank**—Reagent Grade Water containerized during sample collection activities and analyzed at the laboratory. The results are used to determine if sample results may be biased by site environmental factors.
- **Equipment Blank**—Final rinseate collected during sample equipment decontamination and analyzed by the laboratory. The results indicate the effectiveness of the decontamination procedure.
- **Field Duplicate**—An additional sample aliquot or, in some cases, a collocated sample that is collected and analyzed. The results are compared with the original samples as an indication of the overall precision of the entire sampling and analytical process.

5. RESPONSIBILITIES

5.1 Procedure Responsibility

The Field Sampling Discipline Lead is responsible for maintenance, management, and revision of this procedure. Questions, comments, or suggestions regarding this technical SOP should be directed to the Field Sampling Discipline Lead.

5.2 Project Responsibility

Shaw employees performing this task, or any portion thereof, are responsible for meeting the requirements of this procedure. Shaw employees conducting technical review of task performance are also responsible for following appropriate portions of this SOP.

For those projects where the activities of this SOP are conducted, the Project Manager, or designee, is responsible for ensuring that the activities are conducted in accordance with this and other appropriate procedures. Project participants are responsible for documenting information in sufficient detail to provide objective documentation (checkprints, calculations, reports, etc.) that the requirements of this SOP have been met. Such documentation shall be retained as project records.

6. PROCEDURE

6.1 First-Level Review of the Data Package

Verify that the package contains all of the required elements listed in Section 2. If any items are missing, contact the laboratory immediately and correct the situation.

Compare the reported results to the Chain of Custody request, and verify that all expected samples and analyses results were reported. If results are missing, contact the laboratory and correct the situation. If the “missing” data is not available yet, perform partial review of the data provided and hold the package for follow-up once the non-reported results are provided.

6.2 Second-Level Review

Consult the project Chemical Quality Plan (SAP, QAPP, etc.) for information concerning sample types and analysis requirements.

Compare the reported analytes, methods, and detection limits to those in the project plan for the specific analyses. Be sure to account for indicated and reasonable increased reporting limits due to dilutions or sample effects. Address any discrepancies with the laboratory directly.

Compare the results to project action-levels, and circle or otherwise mark all results above the limits.

6.3 QC Level Review

Consult the project Data Usability Review Checklists and/or the project Chemical Quality Plan and evaluate all provided QC results against project acceptance limits.

Mark or flag any results that are outside of the project limits and note on the applicable checklist (if using one).

Also evaluate any Field QC results such as Duplicates and Trip Blanks against requirements and note any issues.

6.4 Usability Review

If all QC results for all samples are within the acceptance ranges, complete the appropriate section of the checklist and then date and sign the completed checklist.

If all QC is acceptable and you are not using a checklist, you must indicate data usability directly on the data package itself or on a separate cover sheet. To do this, date and initial the QC Summary pages and write "QC acceptable data OK for use" on the cover sheet or QC Summary page.

If any QC is non-compliant, review its impact to use as project data by referencing the QC Results Impact Table attached to this SOP and consult with the Qualified Person to determine final acceptability. Note on the Data Report itself or checklist all discrepancies and the reasons for data acceptance, qualification, or rejection. If a Qualified Person has made the decision, this should also be noted.

If any of the data is determined to be unusable, immediately notify the Project Manager and project site personnel.

6.5 Reporting of Usability Review Results

Project personnel must be provided either a spreadsheet summary of the results with an attached, signed and dated Statement of Usability, or the complete Data Package with the project-specific Data Usability Review documentation. At **no time** are results to be communicated verbally.

7. ATTACHMENTS

- Attachment 1, Project QC Impact Table

8. FORMS

None.

Attachment 1
Project QC Impact Table

QC Data Discrepancy	Result Non-detect	Result >10% Below Action-level	Result Within 10% of or Above Action-level	Result Greater than 10% Above Action-level
DISPOSAL				
Trip Blank Contaminated	No effect	No effect	No effect	No effect
LCB Contaminated	No effect on data	No effect on data	No effect unless contamination is >10% of action-level→reject	No effect unless contamination is => the difference between result and action-level
LCS Low Recovery	If MS/MSD are acceptable or Surrogates are acceptable and the RL is at most 20% of action-level→Data accepted	If MS/MSD are acceptable or Surrogates are acceptable→Data accepted Otherwise, flag and qualify that results may in fact be greater than action-level	If MS/MSD are acceptable or Surrogates are acceptable and LCS is within 10% of acceptance limit and result is above action-level→Data accepted Otherwise, flag and qualify result as suspected to be above action-level	No effect on data
LCS High Recovery	No effect on data	No effect on data	If MS/MSD are acceptable or Surrogates are acceptable evaluate potential bias in QC and accept data	No effect on data
Matrix Spike Low %R	If MSD and LCS acceptable and Surrogates or Post-spike within range Data is accepted with precision qualifier	If MSD and LCS acceptable and Surrogates or Post-spike within range Data is accepted with precision qualifier	No effect on data	No effect on data
Matrix Spike High %R	No effect on data	No effect on data	No effect on data	No effect on data
MS/MSD RPD High	No effect on data	No effect on data	No effect on data	No effect on data
Surrogate %R Low	If surrogate %R values are at least 70% of acceptance limit, Data is acceptable	If surrogate %R values are at least 70% of acceptance limit, Data is acceptable	No effect on data	No effect on data
Surrogate %R High	No effect on data	No effect on data	If surrogate %R values are within 30% of acceptance limit→Data is acceptable	No effect on data

QC Data Discrepancy	Result Non-detect	Result >10% Below Action-level	Result Within 10% of or Above Action-level	Result Greater than 10% Above Action-level
REMEDIATION or TREATMENT MONITORING				
Trip Blank Contaminated	No effect	No effect	If TB is greater than 10% of action-level or result → reject data	No effect
Duplicate Precision outside limits	No effect unless Duplicate is either above or within 50% of action-level - in this case qualify sample data and report with Duplicate result as "highest probable value"	No effect unless Duplicate is either above or within 30% of action-level - in this case qualify result as "assumed above action-level"	If Duplicate is either above or within 20% of action-level → qualify result as "assumed above action-level"	No effect-report result even if Duplicate is below action-level
LCB Contaminated	No effect on data	No effect on data	If LCB is greater than 10% of action-level or sample result → Data is unacceptable	No effect on data
LCS Low Recovery	If MS/MSD are acceptable or Surrogates are acceptable → Data accepted	If MS/MSD are acceptable or Surrogates are acceptable → Data accepted	If MS/MSD are acceptable or Surrogates are acceptable → Data accepted	No effect on data
LCS High Recovery	No effect on data	No effect on data	If MS/MSD are acceptable or Surrogates are acceptable evaluate for bias → Data accepted	No effect on data
Matrix Spike Low %R	If %R > 50 and LCS acceptable - Data accepted	If %R > 50 and LCS acceptable - Data accepted	If %R > 50 LCS acceptable → Data accepted (evaluate potential low bias in results below action-level)	No effect
Matrix Spike High %R	No effect on data	No effect on data	If MSD and LCS acceptable and Surrogates or Post-spike within range → Data is accepted with precision qualifier	No effect on data
MS/MSD RPD High	No effect on data unless perceived native concentration in MS or MSD result would be above action-level. In this case, reject data as highly suspect and advise review of sampling and lab sub-sampling procedures	No effect on data unless perceived MS or MSD native concentration would be above action-level. In this case, qualify results as potentially above action-level	If the perceived native result of either the MS or MSD is greater than 110% of action-level → qualify data as being above action-level	No effect on data

QC Data Discrepancy	Result Non-detect	Result >10% Below Action-level	Result Within 10% of or Above Action-level	Result Greater than 10% Above Action-level
Surrogate %R Low	1) If confined to one Surrogate in a fraction, Data is acceptable 2) If surrogate %R values are at least 80% of acceptance limits, Data is acceptable	1) If confined to one Surrogate in a fraction, Data is acceptable 2) If surrogate %R values are at least 80% of acceptance limits, Data is acceptable	No effect on data	No effect on data
Surrogate %R High	No effect on data	No effect on data	If Surrogate %R is greater than 120% of acceptance limit, Data is unacceptable	No effect on data
VERIFICATION or CLOSURE ANALYSIS				
LCB Contaminated	No effect on data Comment LCB contamination	No effect on data Comment LCB contamination	If LCB is greater than 10% of action-level or sample result, Data is unacceptable	If LCB is greater than 10% of action-level or sample result, Data is unacceptable
LCS Low Recovery	If MS/MSD are acceptable or Surrogates are acceptable→Data accepted	If MS/MSD are acceptable or Surrogates are acceptable→Data accepted	If MS/MSD are acceptable or Surrogates are acceptable→Data accepted	If MS/MSD are acceptable or Surrogates are acceptable→Data accepted
LCS High Recovery	No effect on data	No effect on data	If MS/MSD are acceptable or Surrogates are acceptable→Data accepted (evaluate potential bias in reported result)	If MS/MSD are acceptable or Surrogates are acceptable→Data accepted
Matrix Spike Low %R	If MSD and LCS acceptable and Surrogates or Post-spike within range, Data is accepted with precision qualifier	If MSD and LCS acceptable and Surrogates or Post-spike within range, Data is accepted with precision qualifier	If MSD and LCS acceptable and Surrogates or Post-spike within range, Data is accepted with precision qualifier	If MSD and LCS acceptable and Surrogates or Post-spike within range, Data is accepted with precision qualifier
Matrix Spike High %R	If MSD and LCS acceptable and Surrogates or Post-spike within range, Data is accepted with precision qualifier	If MSD and LCS acceptable and Surrogates or Post-spike within range, Data is accepted with precision qualifier	If MSD and LCS acceptable and Surrogates or Post-spike within range, Data is accepted with precision qualifier	If MSD and LCS acceptable and Surrogates or Post-spike within range, Data is accepted with precision qualifier
MS/MSD RPD High	No effect on data	If sample result is greater then 90% of action-level, Data is unacceptable	If RPD is greater than 110% of acceptance limit, Data is unacceptable	If RPD is greater than 110% of acceptance limit, Data is unacceptable

QC Data Discrepancy	Result Non-detect	Result >10% Below Action-level	Result Within 10% of or Above Action-level	Result Greater than 10% Above Action-level
Surrogate %R Low	1) If confined to one Surrogate in a fraction, Data is acceptable 2) If surrogate %R values are at least 80% of acceptance limits, Data is acceptable	1) If confined to one Surrogate in a fraction, Data is acceptable 2) If surrogate %R values are at least 80% of acceptance limits, Data is acceptable	1) If confined to one Surrogate in a fraction, Data is acceptable 2) If surrogate %R values are at least 80% of acceptance limits, Data is acceptable	1) If confined to one Surrogate in a fraction, Data is acceptable 2) If surrogate %R values are at least 80% of acceptance limits, Data is acceptable
Surrogate %R High	1) If confined to one Surrogate in a fraction, Data is acceptable 2) If surrogate %R values are within 20% of acceptance limits, Data is acceptable	1) If confined to one Surrogate in a fraction, Data is acceptable 2) If surrogate %R values are within 20% of acceptance limits and other QC is within acceptance limits, Data is acceptable	If any Surrogate %R is greater than 110% of acceptance limit, Data is unacceptable	1) If confined to one Surrogate in a fraction, Data is acceptable 2) If surrogate %R values are within 20% of acceptance limits, Data is acceptable

STANDARD OPERATING PROCEDURE

Subject: Trowel/Spoon Surface Soil Sampling

1. PURPOSE

The purpose of this document is to provide the methods and procedure for sampling of surface soils using trowels or spoons. Trowels or spoons can be used when matrices are composed of relatively soft and non-cemented formations and to depths of up to 12 inches into the ground surface, dependent on site conditions. Samples for Volatile Organic Compound (VOC) analysis should not be collected via trowel or spoon method. However, a trowel or spoon may be utilized to penetrate to and expose the undisturbed material at the desired depth for sampling by more applicable methods.

2. SCOPE

This procedure is applicable to all Shaw E & I projects where surface soil samples will be collected via trowel or spoon methods.

3. REFERENCES

- U.S. Army Corps of Engineers, 2001, *Requirements for the Preparation of Sampling and Analysis Plans*, Appendix C, Section C.6, EM200-1-3, Washington, D.C.

4. DEFINITIONS

- **Trowel**—A sample collection device with a curved and pointed metal blade attached to a handle. All trace environmental samples should be collected using stainless steel blades.
- **Spoon**—A sample collection device with a round metal blade attached to a handle.
- **Surface Soil**—Soil that is removed from the surface no greater than 6 inches below grade after removing vegetation, rocks, twigs, etc.
- **Weathered Soil**—The top 1/8 to 1/4 inch of soil impacted by heat from sun, rain, or foot traffic that could evaporate, dilute, or otherwise deposit contaminants from an adjacent location, thereby misrepresenting the actual soil characteristic.

5. RESPONSIBILITIES

5.1 Procedure Responsibility

The Field Sampling Discipline Lead is responsible for the maintenance, management, and revision of this procedure. Questions, comments, or suggestions regarding this technical SOP should be directed to the Field Sampling Discipline Lead.

5.2 Project Responsibility

Shaw employees performing this task, or any portion thereof, are responsible for meeting the requirements of this procedure. Shaw employees conducting technical review of task performance are also responsible for following appropriate portions of this SOP.

For those projects where the activities of this SOP are conducted, the Project Manager, or designee, is responsible for ensuring that those activities are conducted in accordance with this and other appropriate procedures. Project participants are responsible for documenting information in sufficient detail to provide objective documentation (checkprints, calculations, reports, etc.) that the requirements of this SOP have been met. Such documentation shall be retained as project records.

6. PROCEDURE

6.1 Equipment

- Decontaminated trowel or spoon, stainless steel construction for trace environmental sampling. If samples will be collected at depth (0-6 inches), the trowel or spoon will require decontamination prior to collection of the targeted-depth sample. Alternatively, a different trowel or spoon can be used to remove the material to the targeted depth and the sample collected using a clean dedicated trowel or spoon.
- Engineers rule or stiff measuring tape
- Decontaminated stainless steel mixing bowl

6.2 Sampling

1. Don a pair of clean gloves.
2. If desired, place plastic sheeting around the targeted location to keep sampled material in place. Use a knife to cut an access hole for the sample location.
3. Remove any surficial debris (e.g. vegetation, rocks, twigs) from the sample location and surrounding area until the soil is exposed. Once exposed, the soil surface is designated as "at grade," or 0 inches.
4. Use a trowel to scrape and remove the top 1/8 to 1/4 inch of weathered soil. (A spoon can be interchanged with trowel).
5. If collecting a sample that includes VOC analysis, collect the VOC sample aliquot first following more applicable methods.
6. With a new trowel, place the point of the blade on the ground. While holding the handle of the trowel, partially rotate the blade in a clockwise/counter-clockwise motion while pushing at a downward angle until the blade is inserted to the required depth or the blade is nearly covered. Be certain that the trowel is not inserted to a depth where the soil will touch the handle or other non-stainless steel portion of the trowel or the sampler's hand.
7. With a prying motion lift up the trowel with soil on the blade and place soil into the stainless steel mixing bowl.
8. Repeat steps 6 and 7 until the required depth of soil is placed into the mixing bowl.
9. Measure the depth of the sample location with a rule or tape to verify the sampling depth and record in the field logbook.
10. Homogenize the non-VOC sample and transfer the sample directly into the sample container(s). Cap the sample container(s), label the containers, complete the documentation, and place the containers into the sample cooler.

7. ATTACHMENTS

None.

8. FORMS

None.

STANDARD OPERATING PROCEDURE

Subject: **Soil Sampling using a Soil Probe or Core-Type Sampler**

1. **PURPOSE**

The purpose of this document is to provide the methods and procedure for sampling of soils and other solids using soil probes and core-type devices. These samplers can be used when matrices are composed of relatively soft and non-cemented formations. They are utilized to collect near-surface core samples and can also be placed into boreholes at specified depths. Soil probe/corer samplers provide an intact depth-specific sample for geotechnical, chemical, radiological, or biological analysis

2. **SCOPE**

This procedure is applicable to all Shaw E & I projects where soil samples will be collected via hand-operated soil probe/corer methods and no project-specific procedure exists. This procedure is not applicable to drilling or direct push methods.

3. **REFERENCES**

- U.S. Army Corps of Engineers, 2001, *Requirements for the Preparation of Sampling and Analysis Plans*, EM-200-1-3.
- American Society for Testing and Materials, *Standard Practice for Soil Investigation and Sampling by Auger Borings*, D1452-80 (re-approved 2000).
- U.S. Environmental Protection Agency, 1994, *Soil Sampling*, EPA/ERT SOP 2012, November.

4. **DEFINITIONS**

- **Soil Corer**—A sample collection device consisting of extension rods, a T-handle, and a sampling head. The sampling head is a thin-walled two-piece metal tube, split lengthwise, into which a metal or plastic sleeve is placed. The tube halves are held together with screw-locked ends, the bottom one having a point. The sleeve fills with material as the sampler is forced downward, allowing for an undisturbed core to be collected
- **Soil Probe**—A core sample collection device consisting of a thin-walled metal tube with a cutting edge on the bottom. The tube is cut-away from its tip to approximately one-third of the way to its top to allow material to enter. The top of a soil probe is removable, and a plastic or metal sleeve is inserted through the top and is held in place by the reduced diameter of the tube at the top of the cutout. Soil probes can be attached to extension rods and T-handles or may be of one-length construction. Samples collected from a soil probe are almost always submitted to the laboratory intact.

5. RESPONSIBILITIES

5.1 Procedure Responsibility

The Field Sampling Discipline Lead is responsible for maintenance, management, and revision of this procedure. Questions, comments, or suggestions regarding this technical SOP should be directed to the Field Sampling Discipline Lead.

5.2 Project Responsibility

Shaw employees performing this task, or any portion thereof, are responsible for meeting the requirements of this procedure. Shaw employees conducting technical review of task performance are also responsible for following appropriate portions of this SOP.

For those projects where the activities of this SOP are conducted, the Project Manager, or designee, is responsible for ensuring that those activities are conducted in accordance with this and other appropriate procedures. Project participants are responsible for documenting information in sufficient detail to provide objective documentation (checkprints, calculations, reports, etc.) that the requirements of this SOP have been met. Such documentation shall be retained as project records.

6. PROCEDURE

The sampling procedure is as follows:

1. Assemble the sampler by inserting the appropriate sample tube and close the ends. If using extension rods, attach the sampler by its top to the bottom rod. Attach the T-handle either to the extension rod or directly to the sampler head.
2. If desired, place plastic sheeting around the targeted location to keep sampled material in place. Use a knife to cut an access hole for the sample location.
3. Don a pair of clean sample gloves.
4. Remove any surficial debris (e.g. vegetation, rocks, twigs) from the sample location and surrounding area.
5. If the sample will be collected from a depth beyond the surface, use a hand-auger to remove the overburden and expose the "target" sample depth. Measure the depth of the hole with a rule or stiff tape to confirm that the target depth has been reached.
6. If the sampling depth is below where the sampling device can be seen while sampling, measure the distance from the tip to top of the sampler and mark the extension rod at this distance plus the depth of the hole with tape as a reference.
7. Change sample gloves just prior to collecting the sample, especially if an auger was used to expose the target depth.
8. To collect the sample using a Soil Corer, place the point of the assembled corer directly on the ground or in the auger hole and, while holding it vertical, push straight down into the soil. Do not twist. A slide hammer may be required for hard or stiff materials.
9. A Soil Probe should be placed into the location and pushed downward with a twisting motion to allow the cutting edge to work. Do not drive or hammer the sampler as this will damage the cutting tip.

10. Continue to force the sampler downward until either the top joint is touching the ground or the reference mark is even with the top of the auger hole. This will ensure that the entire sleeve is filled with material.
11. Extract the sampler by pulling upward with a slight rocking or twisting motion until the head is fully out of the hole.
12. Wipe the sampler head with a cloth or towel and remove it from the T-handle or extension rod.
13. Disassemble the sampler and remove the sleeve. Also perform any field screening desired (e.g., PID screen).
14. For a Soil Probe sample, the sleeve will most likely be submitted intact. Wipe the outside of the sleeve and use a knife to cut off any material sticking from the end so that the ends are even. Place Teflon™ tape over the ends and cap both ends. Be sure to label the top and bottom of the sample interval.
15. A Soil Corer sample may be submitted intact, especially for geotechnical parameters. If this is the case, wipe the outside of the sleeve and use a knife to cut off any material sticking from the end so that the ends are even. Place Teflon™ tape over the ends and cap, labeling the sleeve and marking the top and bottom of the sample interval.
16. If the Soil Corer sample will be aliquotted into other containers, use a knife to split the sleeve lengthwise and remove the top section to expose the sample.
17. If sampling for Volatile Organic Compounds (VOCs), collect sample aliquots from the intact core first using an EnCore™ or other syringe-type device.
18. Place the remaining material directly into sample jars or into a mixing bowl for homogenization and containerization. Cap the sample container(s), label it/them, complete the documentation, and place the sample container(s) into the sample cooler.
19. Decontaminate the sampler.

7. ATTACHMENTS

None.

8. FORMS

None.

STANDARD OPERATING PROCEDURE

Subject: Sediment sampling using a Core Sampler

1. PURPOSE

The purpose of this document is to provide the methods and procedures for collecting sediment samples using sediment/gravity core samplers. These samplers are usually used to collect intact sediment cores in shallow waters. However, they can be mounted onto deep-water drill rigs or similar systems.

2. SCOPE

This procedure is applicable to all Shaw E & I projects where sediment core samples will be collected and no project-specific procedure is in place.

3. REFERENCES

- U.S. Army Corps of Engineers, 2001, *Requirements for the Preparation of Sampling and Analysis Plans*, Appendix C, Section C.6, EM200-1-3, Washington, D.C.
- Wildlife Supply Company (WILDCO) web-site at <http://www.wildco.com/>

4. DEFINITIONS

- **Sediment/Gravity Core Sampler**—A sampling device consisting of a hollow metal tube with a tapered nose-piece collar and a check valve system. The check valve allows water to flow through the sampler body on descent and prevents wash-out of the sample as it is retrieved. The tube is divided lengthwise and accepts a brass or plastic insert sleeve that actually holds the sample. The sampler can be attached to an extension handle and/or drive hammer.

5. RESPONSIBILITIES

5.1 Procedure Responsibility

The Field Sampling Discipline Lead is responsible for maintenance, management, and revision of this procedure. Questions, comments, or suggestions regarding this technical SOP should be directed to the Field Sampling Discipline Lead.

5.2 Project Responsibility

Shaw employees performing this task, or any portion thereof, are responsible for meeting the requirements of this procedure. Shaw employees conducting technical review of task performance are also responsible for following appropriate portions of this SOP.

For those projects where the activities of this SOP are conducted, the Project Manager, or designee, is responsible for ensuring that activities are conducted in accordance with this and other appropriate procedures. Project participants are responsible for documenting information in sufficient detail to provide objective documentation (checkprints, calculations, reports, etc.) that the requirements of this SOP have been met. Such documentation shall be retained as project records.

6. PROCEDURE

*Always tie off to rails or hooks on boats, wear life jackets when appropriate, and abide by all water body safety rules in the project HASP. Sampling that requires either a boat or conveyance across a walkway or other system that exposes personnel to potential of falling in, including sampling for proposal purposes, **must be** performed under an approved HASP and with proper personnel numbers, including a dedicated and unoccupied spotter.*

6.1 Equipment

The following equipment is used for collecting sediment samples with a core sampler:

- Decontaminated commercial sediment/gravity corer with extension handle(s), stainless steel construction for trace environmental sampling
- Brass or plastic sleeves—consult project plan
- Drive hammer, if required
- Plastic sheeting to keep emptying area clean
- Carpenter's chalk or duct/electrical tape
- Plastic or metal shallow pan to empty sampler into

6.2 Sampling Procedure

The procedure for collecting sediment samples with a core sampler is as follows:

1. Don a pair of clean gloves.
2. Place plastic sheeting around the area where the sampler will be emptied to keep sampled material in place.
3. Assemble the sampler by placing an insert sleeve into the tube and attaching the nose-piece and top collar (usually done with screw threads)
4. Attach to an extension or drive hammer system with sufficient length to reach the bottom plus 2- to 3-times the sampler length. Mark the extension at the point equal to the water depth plus the length of the corer tube and nose-piece above the bottom of the corer.
5. Slowly lower the sampler until the bottom is felt.
6. Make sure that the handle/extension is straight up, and push down in a straight direction to force the sampler into the bottom sediment. If using a drive hammer, be sure that the system is straight during each drive.
7. Continue to push/drive the sampler until the mark of the extension is at water level, indicating that the entire sampler has been driven into the sediment.
8. Withdraw the sampler by pulling straight up. It may be necessary to twist slightly while pulling.
9. Retrieve the sampler from the water and place the corer body into the shallow pan.
10. Disassemble the sampler and retrieve the sleeve. Place Teflon™ tape over each end and cap. Label the ends Top and Bottom (T/B).

11. Clean and dry the sleeve; then attach a completed sample label, document the sample, and place it into an appropriate container.

Alternatively, a plastic sleeve can be split lengthwise and then the sample retrieved. Always collect volatile fractions first using a syringe-type or VOC-core sampler.

12. Decontaminate the sampler.

7. ATTACHMENTS

None.

8. FORMS

None.

STANDARD OPERATING PROCEDURE

Subject: Sediment Sampling using Ponar/Ekman Type Systems

1. PURPOSE

The purpose of this document is to provide the methods and procedures for sampling of sediments using clamshell-type sampling devices such as the Ponar and Ekman systems. These sampling systems can be utilized to collect non-core sediment samples. If core samples are desired, alternative methods should be used.

2. SCOPE

This procedure is applicable to all Shaw E & I projects where non-core sediment samples will be collected via clamshell sampling device methods and no project/program specific procedure is in place..

3. REFERENCES

- U.S. Army Corps of Engineers, *Requirements for the Preparation of Sampling and Analysis Plans*, Appendix C, Section C.6, EM200-1-3, Washington, D.C.
- Wildlife Supply Company (WILDCO) web-site at <http://www.wildco.com>.

4. DEFINITIONS

- **Clamshell Device**—A sampling device consisting of spring-loaded jaws that activate either by contact with the bottom or by other means and entrap the collected materials for retrieval. These devices can be operated via hand line or with a winch for deep-water operations.
- **Ekman Sampler**—A type of clamshell device designed for use in soft bottoms. The Ekman sampler rests on the bottom and uses a messenger system to activate the closure spring. The sampler scoops up the material caught between the jaws upon closure.
- **Ponar Sampler**—A type of clamshell device designed for hard and gravelly bottoms. Unlike the Ekman, a Ponar sampler self-activates its closure mechanism after it penetrates into the bottom material. Ponar samplers are heavy (45 lbs.) and require a winch to operate.
- **Messenger**—A metal weight, usually lead, with a hole through its core that is used to activate the spring closure on clamshell devices. The messenger is dropped onto the closure activation mechanism by sliding it down a line. It activates the closure by the force of its weight upon impact.

5. RESPONSIBILITIES

5.1 Procedure Responsibility

The Field Sampling Discipline Lead is responsible for maintenance, management, and revision of this procedure. Questions, comments, or suggestions regarding this technical SOP should be directed to the Field Sampling Discipline Lead.

5.2 Project Responsibility

Shaw E & I employees performing this task, or any portion thereof, are responsible for meeting the requirements of this procedure. Shaw E & I employees conducting technical review of task performance are also responsible for following appropriate portions of this SOP.

For those projects where the activities of this SOP are conducted, the Project Manager, or designee, is responsible for ensuring that activities are conducted in accordance with this and other appropriate procedures. Project participants are responsible for documenting information in sufficient detail to provide objective documentation (checkprints, calculations, reports, etc.) that the requirements of this SOP have been met. Such documentation shall be retained as project records.

6. PROCEDURE

Safety Notes: *These sampling devices are spring activated; they close with great force and are capable of causing injury. Care should be used when opening and securing these devices in the “ready” position. Do not handle by the trip line and always transport in the closed position. Always use proper life-saving equipment and personnel numbers when sampling from a boat or barge. Consult the project Health and Safety Plan for requirements.*

6.1 Equipment

The following equipment should be used when sampling sediments using clamshell-type sampling devices:

- Decontaminated commercial clamshell sampling device, stainless steel construction for trace environmental sampling
- Rope or line with graduations, on winch if required
- Weighted line with graduations to determine depth to bottom, or depth finder if available
- Separate line for messenger if applicable
- Carpenter’s chalk
- Plastic sheeting, to keep emptying area clean
- Plastic or metal shallow pan, to empty sampler into—decontaminated or dedicated
- Stainless steel spoons or scoops—decontaminated or dedicated
- Decontaminated or dedicated stainless steel bowl

6.2 Sampling

The following procedure should be used when sampling sediments using clamshell-type sampling devices:

1. Don a pair of clean gloves.
2. Place plastic sheeting around the area where the sampler will be emptied to keep sampled material in place.
3. Determine the depth to the bottom using the weighted line or depth finder and then mark the sampler’s line at the distance representative of approximately 1m from the bottom with chalk.

4. Attach the line to the sampler and, if applicable, the messenger line. If the messenger has a separate line, make sure it is at least as long as the tag line. *Do not place the messenger on the line at this time.*
5. Carefully open and lock the sampler. From this point on, handle it only by the tag line and take care not to strike it on the release mechanism.
6. Attach the free end of the tag line to a secure holding place to keep from losing the sampler.
7. Being careful not to contact the sampler, slowly lower it into the water until the “1 meter-to-bottom” mark is reached. Make sure that the rope/line does not become entangled.
8. Slow the descent further and continue until the bottom is contacted. Contact with the bottom will be evidenced when the descent stops and slack appears in the line.
9. If sampling with a Ponar, the slack in the line should have activated the closure mechanism. If using a messenger-type system, thread the messenger onto the tag or trip line and allow it to fall and trip the device.
10. Free the device from the bottom by pulling straight up on the tag line, and slowly raise it until it is about 1 to 2 feet from the surface while being careful not to allow the rope/line coils to entangle on anything.
11. Prepare and clear the sample receiving area, and then slowly raise the sampler out of the water.
12. Allow clear water to drain, and swing the sampler onto the pan in the receiving area once the clear liquids have drained. Do not allow the fine particles to exit the sampler also.
13. If a messenger was used, remove it from the line to keep from accidentally tripping the device when retrieving the sample. Carefully open and lock the sampler and allow the sample to fill the pan. Put the sampler aside for cleaning and decontamination.
14. If collecting samples for VOC analysis, these samples should be taken first from the material in the pan using corer or syringe-type methods.
15. The remainder of the sample material should be mixed in the pan and placed into labeled sample containers or other plan-required receptacles using a spoon or scoop.
16. Complete all required documentation, and place the sample into a cooler or other plan-specified container.
17. Decontaminate the sampler on the inside and outside while open and closed to remove all particles. Dry the sampler and return it to its “closed” position when completed.

7. ATTACHMENTS

None.

8. FORMS

None.

STANDARD OPERATING PROCEDURE

Subject: Screening for Metals via X-Ray Fluorescence (XRF) Spectrometry

1. PURPOSE

This procedure is intended to provide general guidance for the analysis of samples using X-ray Fluorescence (XRF) methods. XRF can be used to screen for a variety of metals (Attachment 1) in environmental sample matrices that include soils, air filters, and solid surfaces and materials including dried filter papers, and to screen for lead-based paint (LBP). The XRF technique has been accepted by the U.S. Environmental Protection Agency (EPA) for screening of samples during investigative and remediation actions and is published in SW-846 as Method 6200.

2. SCOPE

This procedure is applicable to all Shaw E & I efforts where metals are to be determined using XRF methods. It is intended to act primarily as a guideline for the use and applicable Quality Control requirements of this technique. This procedure is not intended to replace the applicable manufacturer's information/procedures or those in SW-846, and it also does not present expanded detail on sample preparation. XRF is a surface analysis technique and as such, higher confidence data is achieved when solid samples, especially soil samples, are homogenized and reduced to consistent particle-size mixtures by drying, grinding, and sieving.

Shaw E & I owns a NITON Model XL-703 XRF system with three sources, expanded analyte capabilities, and the filter holder assembly. Contact the Electronics Department in Findlay, Ohio for availability.

3. REFERENCES

- *Field Portable X-ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment, Method 6200, Revision 0, 1998, Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, Third Edition, January.*
- *Instrument Manual for Spectrace Model 9000 XRF, TN Technologies.*
- *Instrument Manual for Niton 700 Series Systems, Niton Inc.*

4. DEFINITIONS

- **X-ray Fluorescence Spectrometry (XRF)**—An analytical technique whereby the sample is exposed to high energy radiation from a radioactive source(s) forcing the ejection of an electron from the target metal specie(s). The vacant space is quickly filled in by another electron from a higher energy shell, with the excess energy released in the form of an X-ray measured in kiloelectron volts (keV) characteristic for a particular metal species.
- **System Blank**—A manufacturer-supplied non-metal implement, such as a Teflon™ or SiO₂ plug, that is placed into the detector and analyzed to evaluate system contamination and scatter.
- **Energy Standard**—A manufacturer-supplied solid metal implement that is analyzed to verify system performance and background correction algorithms.

- **Standard Reference Material (SRM)**—An environmental matrix material containing a known and traceable concentration of analyte(s), usually traceable to the National Institute of Standards and Testing (NIST). It is used to verify analytical accuracy of the system.
- **Site-Specific Calibration**—A calibration process which uses samples from the site itself to develop a calibration curve. Each sample is analyzed by a Definitive method (SW-846) to establish the “true” value. Site-specific calibrations factor in the effect of the site matrix on results and provide better comparison to Definitive data. At least one of the “standards” should be at a concentration near the site action level(s).
- **Sample Cup**—A small plastic or PFE container designed to hold a prepared soil sample and introduce it to the instrument. Sample cups are usually covered with a clear film (Mylar™), which is secured using a plastic ring.

5. RESPONSIBILITIES

5.1 Procedure Responsibility

The Field Sampling Discipline Lead is responsible for maintenance, management, and revision of this procedure. Questions, comments, or suggestions regarding this technical SOP should be directed to the Field Sampling Discipline Lead.

5.2 Project Responsibility

Shaw employees performing this task, or any portion thereof, are responsible for meeting the requirements of this procedure. Shaw employees conducting technical review of task performance are also responsible for following appropriate portions of this SOP.

For those projects where the activities of this SOP are conducted, the Project Manager, or designee, is responsible for ensuring that those activities are conducted in accordance with this and other appropriate procedures. Project participants are responsible for documenting information in sufficient detail to provide objective documentation (i.e. checkprints, calculations, reports, etc.) that the requirements of this SOP have been met. Such documentation shall be retained as project records.

6. PROCEDURE

Safety Notes: *XRF instruments contain radioactive source(s), and the ionizing radiation is hazardous. Do not remove shielding or disassemble instruments beyond the user maintenance dictated in the instrument manual. Never place a hand or other body part in the path of the source, and always operate it with its shield closed, with its safety shutter (if applicable) locked, or with the sensor window held tight against a surface; do not look directly at the beam. Some systems utilize cryogenic cooling systems, and appropriate precautions should be taken during operation.*

These instruments contain regulated radioactive sources and require licensing and specific radioactive licensee procedures for use. In several states, XRF units are considered controlled sources and are subject to state radioactive regulations including specific training for all persons using the instrument, posting of radioactive safety procedures, isolation of work areas, and issuance of state radioactive licenses and permits. Several states require persons transporting, receiving, and/or operating the XRF to attend a Safety Training course every two years for properly handling an instrument containing radioactive sources. Shaw personnel using this type of instrument for the first time must attend the manufacturer's Safety Training course or be trained by a certified Shaw representative. Manufacturers will not send instruments containing regulated radioactive sources to a project site without a competent person as required by their Specific License and General License with an Agreement State where analysis will be performed. For

additional information or assistance in dealing with licensing and/or shipment issues, contact the manufacturer, the leasing agent, or the Radiation Control Officer at the Shaw Oakridge, TN facility.

6.1 General Information

Sensitivity is a function of the instrument count time. Consult the manual to establish a count time that provides the needed sensitivity while allowing for sample throughput efficiency. Typical count times are 60 to 180 seconds.

XRF instruments can be used to screen for metals in filters and filter papers by placing the filter or a portion thereof in the detector and analyzing it. On some systems, a filter holder is used. The filter position, with regards to the detector, should be altered at least three times and the average of the three readings used to determine the result.

Solid surfaces, such as painted walls, are analyzed by holding the unit against the surface and collecting readings or by placing chips into the detector. Again, the average of three readings should be calculated for each result.

Soils can be analyzed by either in situ or ex situ methods depending upon the Data Quality Objectives and data use. In situ analysis can be performed by simply placing the detector onto or over the ground surface and collecting data. However, since XRF is a surface detection technique, it is advisable to at least remove, mix, and sieve a small pile of soil to provide a more consistent matrix. Ex situ analysis involves thorough drying, grinding, mixing, and sieving of the sample and placing it into a sample cup for introduction to the instrument.

XRF instruments are quantitative screening instruments and in most instances provide non-definitive screening data that must be confirmed by definitive methods. They can, however, be utilized for confirmation in well-defined remedial actions governed by detailed approved plans and applicable quality objectives. Use of the method for confirmatory purposes requires enhancements such as site-specific calibration over multiple points, regular QC checks, adjustments of the site-specific curve/Definitive method relationship via split sample analysis, and defined confidence windows for grey-area data. All of these elements should be detailed and defined in a project-specific plan.

6.2 General Operation

- Record all data onto a log sheet or log book.
- Allow the instrument to warm-up for 15 to 30 minutes before use.
- Perform any manufacturer-specified background (scatter) and internal calibration checks using the supplied materials. If the system fails the background check, clean the window and repeat. If the internal calibration check fails, consult the manual to determine how to perform an Energy Calibration. Do not use an instrument that fails either the background check or internal calibration criteria.
- Prior to analyzing any samples, analyze all of the required QC samples and compare to the project criteria. Do not proceed to project samples until QC meets criteria. QC samples in sample cups should be tilted to remix the contents before analysis.
- Proceed with sample analysis. Higher confidence data from soils in sample cups is achieved if each cup is analyzed in duplicate. Tilt the cup to remix the material between each analysis. Report the result as the average of the two values, provided they differ by less than 25%. If they differ by more than 25%, sample preparation methods should be reviewed.

- Analyze a calibration check after every ten sample analyses, following any extended down period, and at the end of the analysis day/shift or as specified in the project plans.
- Download data if required, turn off the analyzer, clean the window, and properly store the instrument when finished. Systems that use batteries should be left on their chargers overnight. Those equipped with safety shutters should always be stored with the shutter in the locked position.

6.3 Quality Control

Quality Control requirements vary depending upon data use and the DQOs necessary and should be defined in a project-specific plan. At a minimum, QC should include analysis of blanks, calibration checks (SRMs or known value samples), and replicate samples.

Confirmatory use requires more extensive QC efforts. A site-specific calibration should be performed by split analyzing prepared samples in duplicate by XRF and off-site definitive methods. The results are used to develop a site-specific XRF/Definitive method correlation and calibration curve. Daily QC should include the analysis of blanks, at least three of the site-specific calibration standards bracketing the expected concentration ranges, replicates, and a check sample or SRM. The correlation should be verified and, if necessary, adjusted on a defined sample analysis or time frequency.

7. ATTACHMENTS

- Attachment 1, List of XRF Analytes and Typical Instrument Detection Limits
- Attachment 2, List of Available SRMs and Properties

8. FORMS

- XRF Calibration Form
- XRF Laboratory Sample Results Form
- XRF Summary of Results

Attachment 1
List of XRF Analytes and Typical Instrument Detection Limits

Element	Typical Reporting Limits for Each Radioactive Source (mg/kg)		
	Fe-55	Cd-109	Am-241
Sulfur	1,500		
Chlorine	450		
Potassium	160		
Calcium	70		
Titanium	55		
Chromium	90	260	
Manganese		205	
Iron		110	
Cobalt		100	
Nickel		65	
Copper		45	
Zinc		35	
Mercury		30	
Arsenic		25	
Selenium		15	
Lead		15	
Rubidium		5	
Strontium		4	
Zirconium		3	
Molybdenum		4	
Cadmium			50
Tin			85
Antimony			45
Barium			30
Silver			9

1-Typically achievable in a clean, silica sand matrix. Actual sample detection limits will be higher due to the sample matrix interferences.

mg/kg - Milligrams per kilogram.

Attachment 2

List of Available SRMs and Properties

Available SRMs / Shaw Owned - Comparisons
[Helps?](#)

SRM ID	Cost	Quan	Metal	Conc	Shaw Current	XRF RL/Source	Yes/OK/No?
2782 - Ind Sludge	\$ 341	70	Ag	30.6	35.3 (2710)	< 9 (Am)	Y
2702 - Marine Sediment	\$ 318	50	As	45.3	22, 23.4, 105, 626, 1550	<20 (Cd)	Y
2780 - Hard Rock	\$ 315	50	As	48.8	22, 23.4, 105, 626, 1550	<20 (Cd)	Y
1648 - Urban PM	\$ 218	2	Cd	75	41.7 (2711)	< 50 (Am)	Y
2702 - Marine Sediment	\$ 318	50	Ni	75.4	(75) (2586)	<65 (Cd)	Y
2781 - Dom Sludge	\$ 294	40	Ni	80.2	(75) (2586)	<65 (Cd)	Y
1648 - Urban PM	\$ 218	2	Ni	82	(75) (2586)	<65 (Cd)	Y
2709 - San J. Soil	\$ 271	50	Ni	88	(75) (2586)	<65 (Cd)	Y
2781 - Dom Sludge	\$ 294	40	Ag	98	35.3 (2710)	< 9 (Am)	Y
2709 - San J. Soil	\$ 271	50	Zn	106	350, 352, 438, 6952	<35 (Cd)	Y
2782 - Ind Sludge	\$ 341	70	Ni	154.1	(75) (2586)	<65 (Cd)	Y
2780 - Hard Rock	\$ 315	50	Sb	160 J	38.4 (2710)	<45 (Am)	Y
2781 - Dom Sludge	\$ 294	40	Pb	202.1	15.6, 161, 432, 1162, 5532	<15 (Cd)	Y
2780 - Hard Rock	\$ 315	50	Cu	215.5	(81), 98.6, 114, 2950	<45 (Cd)	Y
1944 - NY/NJ Sediment	\$ 424	50	Cu	380	(81), 98.6, 114, 2950	<45 (Cd)	Y
1648 - Urban PM	\$ 218	2	Cr	403	301 (2586)	<300 (Cd)	Y
2782 - Ind Sludge	\$ 341	70	Pb	574	15.6, 161, 432, 1162, 5532	<15 (Cd)	Y
1648 - Urban PM	\$ 218	2	Cu	609	(81), 98.6, 114, 2950	<45 (Cd)	Y
2781 - Dom Sludge	\$ 294	40	Cu	627.4	(81), 98.6, 114, 2950	<45 (Cd)	Y
1944 - NY/NJ Sediment	\$ 424	50	Zn	656	350, 352, 438, 6952	<35 (Cd)	Y
2782 - Ind Sludge	\$ 341	70	Zn	1254	350, 352, 438, 6952	<35 (Cd)	Y
2781 - Dom Sludge	\$ 294	40	Zn	1273	350, 352, 438, 6952	<35 (Cd)	Y
2780 - Hard Rock	\$ 315	50	Zn	2570	350, 352, 438, 6952	<35 (Cd)	Y
2587 - Tr Elem in Soil/LBP	\$ 266	55	Pb	3242	15.6, 161, 432, 1162, 5532	<15 (Cd)	Y
1648 - Urban PM	\$ 218	2	Zn	4760	350, 352, 438, 6952	<35 (Cd)	Y
2709 - San J. Soil	\$ 271	50	As	17.7	22, 23.4, 105, 626, 1550	<20 (Cd)	O
2709 - San J. Soil	\$ 271	50	Pb	18.9	15.6, 161, 432, 1162, 5532	<15 (Cd)	O
1944 - NY/NJ Sediment	\$ 424	50	As	18.9	22, 23.4, 105, 626, 1550	<20 (Cd)	O
2780 - Hard Rock	\$ 315	50	Ag	27 J	35.3 (2710)	< 9 (Am)	O
1648 - Urban PM	\$ 218	2	Sb	45 J	38.4 (2710)	<45 (Am)	O
1944 - NY/NJ Sediment	\$ 424	50	Ni	76.1	(75) (2586)	<65 (Cd)	O
1648 - Urban PM	\$ 218	2	As	115	22, 23.4, 105, 626, 1550	<20 (Cd)	O
2702 - Marine Sediment	\$ 318	50	Cu	117.7	(81), 98.6, 114, 2950	<45 (Cd)	O
2702 - Marine Sediment	\$ 318	50	Pb	132.8	15.6, 161, 432, 1162, 5532	<15 (Cd)	O
2782 - Ind Sludge	\$ 341	70	As	166	22, 23.4, 105, 626, 1550	<20 (Cd)	O
1944 - NY/NJ Sediment	\$ 424	50	Cr	266	301 (2586)	<300 (Cd)	O
1944 - NY/NJ Sediment	\$ 424	50	Pb	330	15.6, 161, 432, 1162, 5532	<15 (Cd)	O
2587 - Tr Elem in Soil/LBP	\$ 266	55	Zn	335.8	350, 352, 438, 6952	<35 (Cd)	O
2702 - Marine Sediment	\$ 318	50	Cr	352	301 (2586)	<300 (Cd)	O
2702 - Marine Sediment	\$ 318	50	Zn	485.3	350, 352, 438, 6952	<35 (Cd)	O
2782 - Ind Sludge	\$ 341	70	Cu	2594	(81), 98.6, 114, 2950	<45 (Cd)	O
2780 - Hard Rock	\$ 315	50	Pb	5770	15.6, 161, 432, 1162, 5532	<15 (Cd)	O
1648 - Urban PM	\$ 218	2	Pb	6550	15.6, 161, 432, 1162, 5532	<15 (Cd)	O
2780 - Hard Rock	\$ 315	50	Hg	1 U	32.6, 140	<30 (Cd)	N
2587 - Tr Elem in Soil/LBP	\$ 266	55	Hg	1 U	32.6, 140	<30 (Cd)	N
2702 - Marine Sediment	\$ 318	50	Cd	1 U	41.7 (2711)	< 50 (Am)	N
2702 - Marine Sediment	\$ 318	50	Hg	1 U	32.6, 140	<30 (Cd)	N
2702 - Marine Sediment	\$ 318	50	Ag	1	35.3 (2710)	< 9 (Am)	N
2709 - San J. Soil	\$ 271	50	Cd	1 U	41.7 (2711)	< 50 (Am)	N
2709 - San J. Soil	\$ 271	50	Ag	1 U	35.3 (2710)	< 9 (Am)	N
2782 - Ind Sludge	\$ 341	70	Hg	1.1	32.6, 140	<30 (Cd)	N
2709 - San J. Soil	\$ 271	50	Hg	1.4	32.6, 140	<30 (Cd)	N
2587 - Tr Elem in Soil/LBP	\$ 266	55	Cd	1.92	41.7 (2711)	< 50 (Am)	N
1944 - NY/NJ Sediment	\$ 424	50	Hg	3.4	32.6, 140	<30 (Cd)	N
2781 - Dom Sludge	\$ 294	40	Hg	3.64	32.6, 140	<30 (Cd)	N
2782 - Ind Sludge	\$ 341	70	Cd	4.17	41.7 (2711)	< 50 (Am)	N
1944 - NY/NJ Sediment	\$ 424	50	Sb	5 J	38.4 (2710)	<45 (Am)	N
2702 - Marine Sediment	\$ 318	50	Sb	5.6	38.4 (2710)	<45 (Am)	N

XRF Calibration Form (FS200.1_0)

Site Name - City, State

Project No: Add Number

Contract Title / Client Name

Cost Code: Add Number

1. Initial Calibration Data:

Is XRF warm-up

>15 min? **Yes** **No**

Date: _____

Check XRF clock.

Time: _____

Agree?

Yes **No**

Internal calibration complete?

Yes **No** Comments:

Record energy resolution:

eV

Source Strength: _____ mCi

2. Start-of-Day Calibration:

Note: Cd-109 will be used for Pb, Cu calibration; Am-241 will be used for Sb.

Sources Used (check all that apply):

☒ Cd-109

☐ NA

Fe-55

☐ NA

Am-241

Blank - SiO ₂ , Count: _____ sec			
Element	Conc (ppm)		< DLs Accept?
	Certified	Detected	
Pb	< 25		
Cu	< 50		
Ni	< 70		

Other metals: Zn (<40) _____, As (<30) _____,

Hg(<35) _____, Cr (<300) _____

Low Lead-NIST 2586, Count: _____ sec			
Element	Conc (ppm)		% RPD Accept?
	Certified	Detected	
Pb	432		
Cu	81 *		
Ni	75 *		

Other metals: Zn (352) _____, As (18.9) _____,

Hg(<35) _____, Cr (301) _____

Comments: * Not a NIST certified value.

Moderate Lead-NIST 2711, Count: _____ sec			
Element	Conc (ppm)		% RPD Accept?
	Certified	Detected	
Pb	1,162		
Cu	114		
Ni	< 70		

Other metals: Zn (350.4) _____, As (105) _____,

Hg(<35) _____, Cr (<300) _____

High Lead-NIST 2710, Count: _____ sec			
Element	Conc (ppm)		% RPD Accept?
	Certified	Detected	
Pb	5,532		
Cu	2,950		
Ni	14.3		

Other metals: Zn(6,952) _____, As (626) _____,

Hg(32.6) _____, Cr(<300) _____

3a. Continuing Calibration Data:

Is XRF warm-up

>15 min? **Yes** **No** **N/A**

Date: _____

Check XRF clock.

Time: _____

Agree?

Yes **No**

Internal calibration complete?

Yes **No** Comments:

Record energy resolution:

eV

Source Strength: _____ mCi

3b. Continuing Calibration Data:

Is XRF warm-up

>15 min? **Yes** **No** **N/A**

Date: _____

Check XRF clock.

Time: _____

Agree?

Yes **No**

Internal calibration complete?

Yes **No** Comments:

Record energy resolution:

eV

4. End-of-Day Calibration:

Is XRF warm-up

>15 min? **Yes** **No** **N/A**

Date: _____

Check XRF clock.

Time: _____

Agree?

Yes **No**

Internal calibration complete?

Yes **No** Comments:

Record energy resolution:

eV

Source Strength: _____ mCi

Blank - SiO ₂ , Count: _____ sec			
Element	Conc (ppm)		< DLs Accept?
	Certified	Detected	
Pb	< 25		
Cu	< 50		
Ni	< 70		

Other metals: Zn (<40) _____, As (<30) _____,

Hg(<35) _____, Cr (<300) _____

Low Lead-NIST 2586, Count: _____ sec			
Element	Conc (ppm)		% RPD Accept?
	Certified	Detected	
Pb	432		
Cu	81 *		
Ni	75 *		

Other metals: Zn (352) _____, As (18.9) _____,

Hg(<35) _____, Cr (301) _____

Comments: * Not a NIST certified value.

Moderate Lead-NIST 2711, Count: _____ sec			
Element	Conc (ppm)		% RPD Accept?
	Certified	Detected	
Pb	1,162		
Cu	114		
Ni	< 70		

Other metals: Zn (350.4) _____, As (105) _____,

Hg(<35) _____, Cr (<300) _____

High Lead-NIST 2710, Count: _____ sec			
Element	Conc (ppm)		% RPD Accept?
	Certified	Detected	
Pb	5,532		
Cu	2,950		
Ni	14.3		

Other metals: Zn(6,952) _____, As (626) _____,

Hg(32.6) _____, Cr(<300) _____

Form Completed by:

Signature

Date

XRF Laboratory Sample Results Form

(FS200.2_0)

Site Name - City, State

Project No: Add Number

Contract Title / Client Name

Cost Code: Add Number

1. General Sample Location and Identification:

(circle one): **Floor Sidewall Other**
(if "other" describe): _____

Sample Loc/Description: _____

_____ to _____
(location) (type) (excavation depth, ft)

Location comments: _____ Date: _____ Time: _____

Sample Number:

Associated QC: _____

2. Sample Preparation:

(Check all that apply)

In situ	Drying	Crushing	Coarse Sieve
(pan prep)	(Temp: ____ C for ____ hrs)		(#10, 2mm)

Sample Prep Comments / Tar Observed? _____

3. Analytical Results: (all units are ppm)

Measurement Date: _____

Time: _____

Source(s): Cd-109 ☒ count: _____ sec
Fe-55 ☐ count: N/A sec
Am-241 ☐ count: N/A sec

	Original Analysis	
	Conc	Std Dev
Pb		
As		
Cu		
Zn		

Dup Analysis*		RPD	Comment
Conc	Std Dev	%	

* If performed

Other Metals (Ni, Hg, Cr): _____

Comments: _____

4. Analytical Team:

Form Completed by: _____

Signature

Date



XRF Summary of Results

Site Name - City, State[illegible]

Appendix D

USEPA Method 6200

**Field Portable XRF Spectrometry for the
Determination of Elemental Concentrations in Soil
and Sediment**

METHOD 6200

FIELD PORTABLE X-RAY FLUORESCENCE SPECTROMETRY FOR THE DETERMINATION OF ELEMENTAL CONCENTRATIONS IN SOIL AND SEDIMENT

SW-846 is not intended to be an analytical training manual. Therefore, method procedures are written based on the assumption that they will be performed by analysts who are formally trained in at least the basic principles of chemical analysis and in the use of the subject technology.

In addition, SW-846 methods, with the exception of required method use for the analysis of method-defined parameters, are intended to be guidance methods which contain general information on how to perform an analytical procedure or technique which a laboratory can use as a basic starting point for generating its own detailed Standard Operating Procedure (SOP), either for its own general use or for a specific project application. The performance data included in this method are for guidance purposes only, and are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to the in situ and intrusive analysis of the 26 analytes listed below for soil and sediment samples. Some common elements are not listed in this method because they are considered "light" elements that cannot be detected by field portable x-ray fluorescence (FPXRF). These light elements are: lithium, beryllium, sodium, magnesium, aluminum, silicon, and phosphorus. Most of the analytes listed below are of environmental concern, while a few others have interference effects or change the elemental composition of the matrix, affecting quantitation of the analytes of interest. Generally elements of atomic number 16 or greater can be detected and quantitated by FPXRF. The following RCRA analytes have been determined by this method:

Analytes	CAS Registry No.
Antimony (Sb)	7440-36-0
Arsenic (As)	7440-38-0
Barium (Ba)	7440-39-3
Cadmium (Cd)	7440-43-9
Chromium (Cr)	7440-47-3
Cobalt (Co)	7440-48-4
Copper (Cu)	7440-50-8
Lead (Pb)	7439-92-1
Mercury (Hg)	7439-97-6
Nickel (Ni)	7440-02-0
Selenium (Se)	7782-49-2
Silver (Ag)	7440-22-4
Thallium (Tl)	7440-28-0
Tin (Sn)	7440-31-5

Analytes	CAS Registry No.
Vanadium (V)	7440-62-2
Zinc (Zn)	7440-66-6

In addition, the following non-RCRA analytes have been determined by this method:

Analytes	CAS Registry No.
Calcium (Ca)	7440-70-2
Iron (Fe)	7439-89-6
Manganese (Mn)	7439-96-5
Molybdenum (Mo)	7439-93-7
Potassium (K)	7440-09-7
Rubidium (Rb)	7440-17-7
Strontium (Sr)	7440-24-6
Thorium (Th)	7440-29-1
Titanium (Ti)	7440-32-6
Zirconium (Zr)	7440-67-7

1.2 This method is a screening method to be used with confirmatory analysis using other techniques (e.g., flame atomic absorption spectrometry (FLAA), graphite furnace atomic absorption spectrometry (GFAA), inductively coupled plasma-atomic emission spectrometry, (ICP-AES), or inductively coupled plasma-mass spectrometry, (ICP-MS)). This method's main strength is that it is a rapid field screening procedure. The method's lower limits of detection are typically above the toxicity characteristic regulatory level for most RCRA analytes. However, when the obtainable values for precision, accuracy, and laboratory-established sensitivity of this method meet project-specific data quality objectives (DQOs), FPXRF is a fast, powerful, cost effective technology for site characterization.

1.3 The method sensitivity or lower limit of detection depends on several factors, including the analyte of interest, the type of detector used, the type of excitation source, the strength of the excitation source, count times used to irradiate the sample, physical matrix effects, chemical matrix effects, and interelement spectral interferences. Example lower limits of detection for analytes of interest in environmental applications are shown in Table 1. These limits apply to a clean spiked matrix of quartz sand (silicon dioxide) free of interelement spectral interferences using long (100 -600 second) count times. These sensitivity values are given for guidance only and may not always be achievable, since they will vary depending on the sample matrix, which instrument is used, and operating conditions. A discussion of performance-based sensitivity is presented in Sec. 9.6.

1.4 Analysts should consult the disclaimer statement at the front of the manual and the information in Chapter Two for guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies, and on the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

In addition, analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is *not* mandatory in response to Federal testing requirements. The information contained in this method is provided by EPA as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objectives for the intended application.

1.5 Use of this method is restricted to use by, or under supervision of, personnel appropriately experienced and trained in the use and operation of an XRF instrument. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 The FPXRF technologies described in this method use either sealed radioisotope sources or x-ray tubes to irradiate samples with x-rays. When a sample is irradiated with x-rays, the source x-rays may undergo either scattering or absorption by sample atoms. This latter process is known as the photoelectric effect. When an atom absorbs the source x-rays, the incident radiation dislodges electrons from the innermost shells of the atom, creating vacancies. The electron vacancies are filled by electrons cascading in from outer electron shells. Electrons in outer shells have higher energy states than inner shell electrons, and the outer shell electrons give off energy as they cascade down into the inner shell vacancies. This rearrangement of electrons results in emission of x-rays characteristic of the given atom. The emission of x-rays, in this manner, is termed x-ray fluorescence.

Three electron shells are generally involved in emission of x-rays during FPXRF analysis of environmental samples. The three electron shells include the K, L, and M shells. A typical emission pattern, also called an emission spectrum, for a given metal has multiple intensity peaks generated from the emission of K, L, or M shell electrons. The most commonly measured x-ray emissions are from the K and L shells; only metals with an atomic number greater than 57 have measurable M shell emissions.

Each characteristic x-ray line is defined with the letter K, L, or M, which signifies which shell had the original vacancy and by a subscript alpha (α), beta (β), or gamma (γ) etc., which indicates the higher shell from which electrons fell to fill the vacancy and produce the x-ray. For example, a K_α line is produced by a vacancy in the K shell filled by an L shell electron, whereas a K_β line is produced by a vacancy in the K shell filled by an M shell electron. The K_α transition is on average 6 to 7 times more probable than the K_β transition; therefore, the K_α line is approximately 7 times more intense than the K_β line for a given element, making the K_α line the choice for quantitation purposes.

The K lines for a given element are the most energetic lines and are the preferred lines for analysis. For a given atom, the x-rays emitted from L transitions are always less energetic than those emitted from K transitions. Unlike the K lines, the main L emission lines (L_α and L_β) for an element are of nearly equal intensity. The choice of one or the other depends on what interfering element lines might be present. The L emission lines are useful for analyses involving elements of atomic number (Z) 58 (cerium) through 92 (uranium).

An x-ray source can excite characteristic x-rays from an element only if the source energy is greater than the absorption edge energy for the particular line group of the element, that is, the K absorption edge, L absorption edge, or M absorption edge energy. The absorption edge energy is somewhat greater than the corresponding line energy. Actually, the K absorption edge energy is approximately the sum of the K, L, and M line energies of the particular element, and the L absorption edge energy is approximately the sum of the L and M line energies. FPXRF is more sensitive to an element with an absorption edge energy close to but less than

the excitation energy of the source. For example, when using a cadmium-109 source, which has an excitation energy of 22.1 kiloelectron volts (keV), FPXRF would exhibit better sensitivity for zirconium which has a K line energy of 15.77 keV than to chromium, which has a K line energy of 5.41 keV.

2.2 Under this method, inorganic analytes of interest are identified and quantitated using a field portable energy-dispersive x-ray fluorescence spectrometer. Radiation from one or more radioisotope sources or an electrically excited x-ray tube is used to generate characteristic x-ray emissions from elements in a sample. Up to three sources may be used to irradiate a sample. Each source emits a specific set of primary x-rays that excite a corresponding range of elements in a sample. When more than one source can excite the element of interest, the source is selected according to its excitation efficiency for the element of interest.

For measurement, the sample is positioned in front of the probe window. This can be done in two manners using FPXRF instruments, specifically, in situ or intrusive. If operated in the in situ mode, the probe window is placed in direct contact with the soil surface to be analyzed. When an FPXRF instrument is operated in the intrusive mode, a soil or sediment sample must be collected, prepared, and placed in a sample cup. The sample cup is then placed on top of the window inside a protective cover for analysis.

Sample analysis is then initiated by exposing the sample to primary radiation from the source. Fluorescent and backscattered x-rays from the sample enter through the detector window and are converted into electric pulses in the detector. The detector in FPXRF instruments is usually either a solid-state detector or a gas-filled proportional counter. Within the detector, energies of the characteristic x-rays are converted into a train of electric pulses, the amplitudes of which are linearly proportional to the energy of the x-rays. An electronic multichannel analyzer (MCA) measures the pulse amplitudes, which is the basis of qualitative x-ray analysis. The number of counts at a given energy per unit of time is representative of the element concentration in a sample and is the basis for quantitative analysis. Most FPXRF instruments are menu-driven from software built into the units or from personal computers (PC).

The measurement time of each source is user-selectable. Shorter source measurement times (30 seconds) are generally used for initial screening and hot spot delineation, and longer measurement times (up to 300 seconds) are typically used to meet higher precision and accuracy requirements.

FPXRF instruments can be calibrated using the following methods: internally using fundamental parameters determined by the manufacturer, empirically based on site-specific calibration standards (SSCS), or based on Compton peak ratios. The Compton peak is produced by backscattering of the source radiation. Some FPXRF instruments can be calibrated using multiple methods.

3.0 DEFINITIONS

3.1 FPXRF -- Field portable x-ray fluorescence.

3.2 MCA -- Multichannel analyzer for measuring pulse amplitude.

3.3 SSCS -- Site-specific calibration standards.

3.4 FP -- Fundamental parameter.

3.5 ROI -- Region of interest.

3.6 SRM -- Standard reference material; a standard containing certified amounts of metals in soil or sediment.

3.7 eV -- Electron volt; a unit of energy equivalent to the amount of energy gained by an electron passing through a potential difference of one volt.

3.8 Refer to Chapter One, Chapter Three, and the manufacturer's instructions for other definitions that may be relevant to this procedure.

4.0 INTERFERENCES

4.1 The total method error for FPXRF analysis is defined as the square root of the sum of squares of both instrument precision and user- or application-related error. Generally, instrument precision is the least significant source of error in FPXRF analysis. User- or application-related error is generally more significant and varies with each site and method used. Some sources of interference can be minimized or controlled by the instrument operator, but others cannot. Common sources of user- or application-related error are discussed below.

4.2 Physical matrix effects result from variations in the physical character of the sample. These variations may include such parameters as particle size, uniformity, homogeneity, and surface condition. For example, if any analyte exists in the form of very fine particles in a coarser-grained matrix, the analyte's concentration measured by the FPXRF will vary depending on how fine particles are distributed within the coarser-grained matrix. If the fine particles "settle" to the bottom of the sample cup (i.e., against the cup window), the analyte concentration measurement will be higher than if the fine particles are not mixed in well and stay on top of the coarser-grained particles in the sample cup. One way to reduce such error is to grind and sieve all soil samples to a uniform particle size thus reducing sample-to-sample particle size variability. Homogeneity is always a concern when dealing with soil samples. Every effort should be made to thoroughly mix and homogenize soil samples before analysis. Field studies have shown heterogeneity of the sample generally has the largest impact on comparability with confirmatory samples.

4.3 Moisture content may affect the accuracy of analysis of soil and sediment sample analyses. When the moisture content is between 5 and 20 percent, the overall error from moisture may be minimal. However, moisture content may be a major source of error when analyzing samples of surface soil or sediment that are saturated with water. This error can be minimized by drying the samples in a convection or toaster oven. Microwave drying is not recommended because field studies have shown that microwave drying can increase variability between FPXRF data and confirmatory analysis and because metal fragments in the sample can cause arcing to occur in a microwave.

4.4 Inconsistent positioning of samples in front of the probe window is a potential source of error because the x-ray signal decreases as the distance from the radioactive source increases. This error is minimized by maintaining the same distance between the window and each sample. For the best results, the window of the probe should be in direct contact with the sample, which means that the sample should be flat and smooth to provide a good contact surface.

4.5 Chemical matrix effects result from differences in the concentrations of interfering elements. These effects occur as either spectral interferences (peak overlaps) or as x-ray absorption and enhancement phenomena. Both effects are common in soils contaminated with heavy metals. As examples of absorption and enhancement effects; iron (Fe) tends to absorb copper (Cu) x-rays, reducing the intensity of the Cu measured by the detector, while chromium (Cr) will be enhanced at the expense of Fe because the absorption edge of Cr is slightly lower in energy than the fluorescent peak of iron. The effects can be corrected mathematically through the use of fundamental parameter (FP) coefficients. The effects also can be compensated for using SSCS, which contain all the elements present on site that can interfere with one another.

4.6 When present in a sample, certain x-ray lines from different elements can be very close in energy and, therefore, can cause interference by producing a severely overlapped spectrum. The degree to which a detector can resolve the two different peaks depends on the energy resolution of the detector. If the energy difference between the two peaks in electron volts is less than the resolution of the detector in electron volts, then the detector will not be able to fully resolve the peaks.

The most common spectrum overlaps involve the K_{β} line of element Z-1 with the K_{α} line of element Z. This is called the K_{α}/K_{β} interference. Because the $K_{\alpha}:K_{\beta}$ intensity ratio for a given element usually is about 7:1, the interfering element, Z-1, must be present at large concentrations to cause a problem. Two examples of this type of spectral interference involve the presence of large concentrations of vanadium (V) when attempting to measure Cr or the presence of large concentrations of Fe when attempting to measure cobalt (Co). The V K_{α} and K_{β} energies are 4.95 and 5.43 keV, respectively, and the Cr K_{α} energy is 5.41 keV. The Fe K_{α} and K_{β} energies are 6.40 and 7.06 keV, respectively, and the Co K_{α} energy is 6.92 keV. The difference between the V K_{β} and Cr K_{α} energies is 20 eV, and the difference between the Fe K_{β} and the Co K_{α} energies is 140 eV. The resolution of the highest-resolution detectors in FPXRF instruments is 170 eV. Therefore, large amounts of V and Fe will interfere with quantitation of Cr or Co, respectively. The presence of Fe is a frequent problem because it is often found in soils at tens of thousands of parts per million (ppm).

4.7 Other interferences can arise from K/L, K/M, and L/M line overlaps, although these overlaps are less common. Examples of such overlap involve arsenic (As) K_{α} /lead (Pb) L_{α} and sulfur (S) K_{α} /Pb M_{α} . In the As/Pb case, Pb can be measured from the Pb L_{β} line, and As can be measured from either the As K_{α} or the As K_{β} line; in this way the interference can be corrected. If the As K_{β} line is used, sensitivity will be decreased by a factor of two to five times because it is a less intense line than the As K_{α} line. If the As K_{α} line is used in the presence of Pb, mathematical corrections within the instrument software can be used to subtract out the Pb interference. However, because of the limits of mathematical corrections, As concentrations cannot be efficiently calculated for samples with Pb:As ratios of 10:1 or more. This high ratio of Pb to As may result in reporting of a "nondetect" or a "less than" value (e.g., <300 ppm) for As, regardless of the actual concentration present.

No instrument can fully compensate for this interference. It is important for an operator to understand this limitation of FPXRF instruments and consult with the manufacturer of the FPXRF instrument to evaluate options to minimize this limitation. The operator's decision will be based on action levels for metals in soil established for the site, matrix effects, capabilities of the instrument, data quality objectives, and the ratio of lead to arsenic known to be present at the site. If a site is encountered that contains lead at concentrations greater than ten times the concentration of arsenic it is advisable that all critical soil samples be sent off site for confirmatory analysis using other techniques (e.g., flame atomic absorption spectrometry (FLAA), graphite furnace atomic absorption spectrometry (GFAA), inductively coupled plasma-

atomic emission spectrometry, (ICP-AES), or inductively coupled plasma-mass spectrometry, (ICP-MS)).

4.8 If SSCS are used to calibrate an FPXRF instrument, the samples collected must be representative of the site under investigation. Representative soil sampling ensures that a sample or group of samples accurately reflects the concentrations of the contaminants of concern at a given time and location. Analytical results for representative samples reflect variations in the presence and concentration ranges of contaminants throughout a site. Variables affecting sample representativeness include differences in soil type, contaminant concentration variability, sample collection and preparation variability, and analytical variability, all of which should be minimized as much as possible.

4.9 Soil physical and chemical effects may be corrected using SSCS that have been analyzed by inductively coupled plasma (ICP) or atomic absorption (AA) methods. However, a major source of error can be introduced if these samples are not representative of the site or if the analytical error is large. Another concern is the type of digestion procedure used to prepare the soil samples for the reference analysis. Analytical results for the confirmatory method will vary depending on whether a partial digestion procedure, such as Method 3050, or a total digestion procedure, such as Method 3052, is used. It is known that depending on the nature of the soil or sediment, Method 3050 will achieve differing extraction efficiencies for different analytes of interest. The confirmatory method should meet the project-specific data quality objectives (DQOs).

XRF measures the total concentration of an element; therefore, to achieve the greatest comparability of this method with the reference method (reduced bias), a total digestion procedure should be used for sample preparation. However, in the study used to generate the performance data for this method (see Table 8), the confirmatory method used was Method 3050, and the FPXRF data compared very well with regression correlation coefficients (r often exceeding 0.95, except for barium and chromium). The critical factor is that the digestion procedure and analytical reference method used should meet the DQOs of the project and match the method used for confirmation analysis.

4.10 Ambient temperature changes can affect the gain of the amplifiers producing instrument drift. Gain or drift is primarily a function of the electronics (amplifier or preamplifier) and not the detector as most instrument detectors are cooled to a constant temperature. Most FPXRF instruments have a built-in automatic gain control. If the automatic gain control is allowed to make periodic adjustments, the instrument will compensate for the influence of temperature changes on its energy scale. If the FPXRF instrument has an automatic gain control function, the operator will not have to adjust the instrument's gain unless an error message appears. If an error message appears, the operator should follow the manufacturer's procedures for troubleshooting the problem. Often, this involves performing a new energy calibration. The performance of an energy calibration check to assess drift is a quality control measure discussed in Sec. 9.2.

If the operator is instructed by the manufacturer to manually conduct a gain check because of increasing or decreasing ambient temperature, it is standard to perform a gain check after every 10 to 20 sample measurements or once an hour whichever is more frequent. It is also suggested that a gain check be performed if the temperature fluctuates more than 10° F. The operator should follow the manufacturer's recommendations for gain check frequency.

5.0 SAFETY

5.1 This method does not address all safety issues associated with its use. The user is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals listed in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

NOTE: No MSDS applies directly to the radiation-producing instrument because that is covered under the Nuclear Regulatory Commission (NRC) or applicable state regulations.

5.2 Proper training for the safe operation of the instrument and radiation training should be completed by the analyst prior to analysis. Radiation safety for each specific instrument can be found in the operator's manual. Protective shielding should never be removed by the analyst or any personnel other than the manufacturer. The analyst should be aware of the local state and national regulations that pertain to the use of radiation-producing equipment and radioactive materials with which compliance is required. There should be a person appointed within the organization that is solely responsible for properly instructing all personnel, maintaining inspection records, and monitoring x-ray equipment at regular intervals.

Licenses for radioactive materials are of two types, specifically: (1) a general license which is usually initiated by the manufacturer for receiving, acquiring, owning, possessing, using, and transferring radioactive material incorporated in a device or equipment, and (2) a specific license which is issued to named persons for the operation of radioactive instruments as required by local, state, or federal agencies. A copy of the radioactive material license (for specific licenses only) and leak tests should be present with the instrument at all times and available to local and national authorities upon request.

X-ray tubes do not require radioactive material licenses or leak tests, but do require approvals and licenses which vary from state to state. In addition, fail-safe x-ray warning lights should be illuminated whenever an x-ray tube is energized. Provisions listed above concerning radiation safety regulations, shielding, training, and responsible personnel apply to x-ray tubes just as to radioactive sources. In addition, a log of the times and operating conditions should be kept whenever an x-ray tube is energized. An additional hazard present with x-ray tubes is the danger of electric shock from the high voltage supply, however, if the tube is properly positioned within the instrument, this is only a negligible risk. Any instrument (x-ray tube or radioisotope based) is capable of delivering an electric shock from the basic circuitry when the system is inappropriately opened.

5.3 Radiation monitoring equipment should be used with the handling and operation of the instrument. The operator and the surrounding environment should be monitored continually for analyst exposure to radiation. Thermal luminescent detectors (TLD) in the form of badges and rings are used to monitor operator radiation exposure. The TLDs or badges should be worn in the area of maximum exposure. The maximum permissible whole-body dose from occupational exposure is 5 Roentgen Equivalent Man (REM) per year. Possible exposure pathways for radiation to enter the body are ingestion, inhaling, and absorption. The best precaution to prevent radiation exposure is distance and shielding.

6.0 EQUIPMENT AND SUPPLIES

The mention of trade names or commercial products in this manual is for illustrative purposes only, and does not constitute an EPA endorsement or exclusive recommendation for

use. The products and instrument settings cited in SW-846 methods represent those products and settings used during method development or subsequently evaluated by the Agency. Glassware, reagents, supplies, equipment, and settings other than those listed in this manual may be employed provided that method performance appropriate for the intended application has been demonstrated and documented.

6.1 FPXRF spectrometer -- An FPXRF spectrometer consists of four major components: (1) a source that provides x-rays; (2) a sample presentation device; (3) a detector that converts x-ray-generated photons emitted from the sample into measurable electronic signals; and (4) a data processing unit that contains an emission or fluorescence energy analyzer, such as an MCA, that processes the signals into an x-ray energy spectrum from which elemental concentrations in the sample may be calculated, and a data display and storage system. These components and additional, optional items, are discussed below.

6.1.1 Excitation sources -- FPXRF instruments use either a sealed radioisotope source or an x-ray tube to provide the excitation source. Many FPXRF instruments use sealed radioisotope sources to produce x-rays in order to irradiate samples. The FPXRF instrument may contain between one and three radioisotope sources. Common radioisotope sources used for analysis for metals in soils are iron Fe-55 (^{55}Fe), cadmium Cd-109 (^{109}Cd), americium Am-241 (^{241}Am), and curium Cm-244 (^{244}Cm). These sources may be contained in a probe along with a window and the detector; the probe may be connected to a data reduction and handling system by means of a flexible cable. Alternatively, the sources, window, and detector may be included in the same unit as the data reduction and handling system.

The relative strength of the radioisotope sources is measured in units of millicuries (mCi). All other components of the FPXRF system being equal, the stronger the source, the greater the sensitivity and precision of a given instrument. Radioisotope sources undergo constant decay. In fact, it is this decay process that emits the primary x-rays used to excite samples for FPXRF analysis. The decay of radioisotopes is measured in "half-lives." The half-life of a radioisotope is defined as the length of time required to reduce the radioisotopes strength or activity by half. Developers of FPXRF technologies recommend source replacement at regular intervals based on the source's half-life. This is due to the ever increasing time required for the analysis rather than a decrease in instrument performance. The characteristic x-rays emitted from each of the different sources have energies capable of exciting a certain range of analytes in a sample. Table 2 summarizes the characteristics of four common radioisotope sources.

X-ray tubes have higher radiation output, no intrinsic lifetime limit, produce constant output over their lifetime, and do not have the disposal problems of radioactive sources but are just now appearing in FPXRF instruments. An electrically-excited x-ray tube operates by bombarding an anode with electrons accelerated by a high voltage. The electrons gain an energy in electron volts equal to the accelerating voltage and can excite atomic transitions in the anode, which then produces characteristic x-rays. These characteristic x-rays are emitted through a window which contains the vacuum necessary for the electron acceleration. An important difference between x-ray tubes and radioactive sources is that the electrons which bombard the anode also produce a continuum of x-rays across a broad range of energies in addition to the characteristic x-rays. This continuum is weak compared to the characteristic x-rays but can provide substantial excitation since it covers a broad energy range. It has the undesired property of producing background in the spectrum near the analyte x-ray lines when it is scattered by the sample. For this reason a filter is often used between the x-ray tube and the sample to suppress the continuum radiation while passing the characteristic x-rays from the anode. This filter is sometimes incorporated into the window of the x-ray tube. The choice of

accelerating voltage is governed both by the anode material, since the electrons must have sufficient energy to excite the anode, which requires a voltage greater than the absorption edge of the anode material and by the instrument's ability to cool the x-ray tube. The anode is most efficiently excited by voltages 2 to 2.5 times the edge energy (most x-rays per unit power to the tube), although voltages as low as 1.5 times the absorption edge energy will work. The characteristic x-rays emitted by the anode are capable of exciting a range of elements in the sample just as with a radioactive source. Table 3 gives the recommended operating voltages and the sample elements excited for some common anodes.

6.1.2 Sample presentation device -- FPXRF instruments can be operated in two modes: in situ and intrusive. If operated in the in situ mode, the probe window is placed in direct contact with the soil surface to be analyzed. When an FPXRF instrument is operated in the intrusive mode, a soil or sediment sample must be collected, prepared, and placed in a sample cup. For FPXRF instruments operated in the intrusive mode, the probe may be rotated so that the window faces either upward or downward. A protective sample cover is placed over the window, and the sample cup is placed on top of the window inside the protective sample cover for analysis.

6.1.3 Detectors -- The detectors in the FPXRF instruments can be either solid-state detectors or gas-filled, proportional counter detectors. Common solid-state detectors include mercuric iodide (HgI_2), silicon pin diode and lithium-drifted silicon $\text{Si}(\text{Li})$. The HgI_2 detector is operated at a moderately subambient temperature controlled by a low power thermoelectric cooler. The silicon pin diode detector also is cooled via the thermoelectric Peltier effect. The $\text{Si}(\text{Li})$ detector must be cooled to at least -90°C either with liquid nitrogen or by thermoelectric cooling via the Peltier effect. Instruments with a $\text{Si}(\text{Li})$ detector have an internal liquid nitrogen dewar with a capacity of 0.5 to 1.0 L. Proportional counter detectors are rugged and lightweight, which are important features of a field portable detector. However, the resolution of a proportional counter detector is not as good as that of a solid-state detector. The energy resolution of a detector for characteristic x-rays is usually expressed in terms of full width at half-maximum (FWHM) height of the manganese K_α peak at 5.89 keV. The typical resolutions of the above mentioned detectors are as follows: HgI_2 –270 eV; silicon pin diode–250 eV; $\text{Si}(\text{Li})$ –170 eV; and gas-filled, proportional counter–750 eV.

During operation of a solid-state detector, an x-ray photon strikes a biased, solid-state crystal and loses energy in the crystal by producing electron-hole pairs. The electric charge produced is collected and provides a current pulse that is directly proportional to the energy of the x-ray photon absorbed by the crystal of the detector. A gas-filled, proportional counter detector is an ionization chamber filled with a mixture of noble and other gases. An x-ray photon entering the chamber ionizes the gas atoms. The electric charge produced is collected and provides an electric signal that is directly proportional to the energy of the x-ray photon absorbed by the gas in the detector.

6.1.4 Data processing units -- The key component in the data processing unit of an FPXRF instrument is the MCA. The MCA receives pulses from the detector and sorts them by their amplitudes (energy level). The MCA counts pulses per second to determine the height of the peak in a spectrum, which is indicative of the target analyte's concentration. The spectrum of element peaks are built on the MCA. The MCAs in FPXRF instruments have from 256 to 2,048 channels. The concentrations of target analytes are usually shown in ppm on a liquid crystal display (LCD) in the instrument. FPXRF instruments can store both spectra and from 3,000 to 5,000 sets of numerical analytical results. Most FPXRF instruments are menu-driven from software built into the

units or from PCs. Once the data-storage memory of an FPXRF unit is full or at any other time, data can be downloaded by means of an RS-232 port and cable to a PC.

6.2 Spare battery and battery charger.

6.3 Polyethylene sample cups -- 31 to 40 mm in diameter with collar, or equivalent (appropriate for FPXRF instrument).

6.4 X-ray window film -- Mylar™, Kapton™, Spectrolene™, polypropylene, or equivalent; 2.5 to 6.0 µm thick.

6.5 Mortar and pestle -- Glass, agate, or aluminum oxide; for grinding soil and sediment samples.

6.6 Containers -- Glass or plastic to store samples.

6.7 Sieves -- 60-mesh (0.25 mm), stainless-steel, Nylon, or equivalent for preparing soil and sediment samples.

6.8 Trowels -- For smoothing soil surfaces and collecting soil samples.

6.9 Plastic bags -- Used for collection and homogenization of soil samples.

6.10 Drying oven -- Standard convection or toaster oven, for soil and sediment samples that require drying.

7.0 REAGENTS AND STANDARDS

7.1 Reagent grade chemicals must be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Pure element standards -- Each pure, single-element standard is intended to produce strong characteristic x-ray peaks of the element of interest only. Other elements present must not contribute to the fluorescence spectrum. A set of pure element standards for commonly sought analytes is supplied by the instrument manufacturer, if designated for the instrument; not all instruments require the pure element standards. The standards are used to set the region of interest (ROI) for each element. They also can be used as energy calibration and resolution check samples.

7.3 Site-specific calibration standards -- Instruments that employ fundamental parameters (FP) or similar mathematical models in minimizing matrix effects may not require SSCS. If the FP calibration model is to be optimized or if empirical calibration is necessary, then SSCSs must be collected, prepared, and analyzed.

7.3.1 The SSCS must be representative of the matrix to be analyzed by FPXRF. These samples must be well homogenized. A minimum of 10 samples spanning the concentration ranges of the analytes of interest and of the interfering elements must be obtained from the site. A sample size of 4 to 8 ounces is recommended, and standard glass sampling jars should be used.

7.3.2 Each sample should be oven-dried for 2 to 4 hr at a temperature of less than 150 °C. If mercury is to be analyzed, a separate sample portion should be dried at ambient temperature as heating may volatilize the mercury. When the sample is dry, all large, organic debris and nonrepresentative material, such as twigs, leaves, roots, insects, asphalt, and rock should be removed. The sample should be homogenized (see Sec. 7.3.3) and then a representative portion ground with a mortar and pestle or other mechanical means, prior to passing through a 60-mesh sieve. Only the coarse rock fraction should remain on the screen.

7.3.3 The sample should be homogenized by using a riffle splitter or by placing 150 to 200 g of the dried, sieved sample on a piece of kraft or butcher paper about 1.5 by 1.5 feet in size. Each corner of the paper should be lifted alternately, rolling the soil over on itself and toward the opposite corner. The soil should be rolled on itself 20 times. Approximately 5 g of the sample should then be removed and placed in a sample cup for FPXRF analysis. The rest of the prepared sample should be sent off site for ICP or AA analysis. The method use for confirmatory analysis should meet the data quality objectives of the project.

7.4 Blank samples -- The blank samples should be from a "clean" quartz or silicon dioxide matrix that is free of any analytes at concentrations above the established lower limit of detection. These samples are used to monitor for cross-contamination and laboratory-induced contaminants or interferences.

7.5 Standard reference materials -- Standard reference materials (SRMs) are standards containing certified amounts of metals in soil or sediment. These standards are used for accuracy and performance checks of FPXRF analyses. SRMs can be obtained from the National Institute of Standards and Technology (NIST), the U.S. Geological Survey (USGS), the Canadian National Research Council, and the national bureau of standards in foreign nations. Pertinent NIST SRMs for FPXRF analysis include 2704, Buffalo River Sediment; 2709, San Joaquin Soil; and 2710 and 2711, Montana Soil. These SRMs contain soil or sediment from actual sites that has been analyzed using independent inorganic analytical methods by many different laboratories. When these SRMs are unavailable, alternate standards may be used (e.g., NIST 2702).

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

Sample handling and preservation procedures used in FPXRF analyses should follow the guidelines in Chapter Three, "Inorganic Analytes."

9.0 QUALITY CONTROL

9.1 Follow the manufacturer's instructions for the quality control procedures specific to use of the testing product. Refer to Chapter One for additional guidance on quality assurance (QA) and quality control (QC) protocols. Any effort involving the collection of analytical data should include development of a structured and systematic planning document, such as a Quality Assurance Project Plan (QAPP) or a Sampling and Analysis Plan (SAP), which translates project objectives and specifications into directions for those that will implement the project and assess the results.

9.2 Energy calibration check -- To determine whether an FPXRF instrument is operating within resolution and stability tolerances, an energy calibration check should be run. The energy calibration check determines whether the characteristic x-ray lines are shifting,

which would indicate drift within the instrument. As discussed in Sec. 4.10, this check also serves as a gain check in the event that ambient temperatures are fluctuating greatly (more than 10 °F).

9.2.1 The energy calibration check should be run at a frequency consistent with manufacturer's recommendations. Generally, this would be at the beginning of each working day, after the batteries are changed or the instrument is shut off, at the end of each working day, and at any other time when the instrument operator believes that drift is occurring during analysis. A pure element such as iron, manganese, copper, or lead is often used for the energy calibration check. A manufacturer-recommended count time per source should be used for the check.

9.2.2 The instrument manufacturer's manual specifies the channel or kiloelectron volt level at which a pure element peak should appear and the expected intensity of the peak. The intensity and channel number of the pure element as measured using the source should be checked and compared to the manufacturer's recommendation. If the energy calibration check does not meet the manufacturer's criteria, then the pure element sample should be repositioned and reanalyzed. If the criteria are still not met, then an energy calibration should be performed as described in the manufacturer's manual. With some FPXRF instruments, once a spectrum is acquired from the energy calibration check, the peak can be optimized and realigned to the manufacturer's specifications using their software.

9.3 Blank samples -- Two types of blank samples should be analyzed for FPXRF analysis, specifically, instrument blanks and method blanks.

9.3.1 An instrument blank is used to verify that no contamination exists in the spectrometer or on the probe window. The instrument blank can be silicon dioxide, a polytetrafluoroethylene (PTFE) block, a quartz block, "clean" sand, or lithium carbonate. This instrument blank should be analyzed on each working day before and after analyses are conducted and once per every twenty samples. An instrument blank should also be analyzed whenever contamination is suspected by the analyst. The frequency of analysis will vary with the data quality objectives of the project. A manufacturer-recommended count time per source should be used for the blank analysis. No element concentrations above the established lower limit of detection should be found in the instrument blank. If concentrations exceed these limits, then the probe window and the check sample should be checked for contamination. If contamination is not a problem, then the instrument must be "zeroed" by following the manufacturer's instructions.

9.3.2 A method blank is used to monitor for laboratory-induced contaminants or interferences. The method blank can be "clean" silica sand or lithium carbonate that undergoes the same preparation procedure as the samples. A method blank must be analyzed at least daily. The frequency of analysis will depend on the data quality objectives of the project. If the method blank does not contain the target analyte at a level that interferes with the project-specific data quality objectives then the method blank would be considered acceptable. In the absence of project-specific data quality objectives, if the blank is less than the lowest level of detection or less than 10% of the lowest sample concentration for the analyte, whichever is greater, then the method blank would be considered acceptable. If the method blank cannot be considered acceptable, the cause of the problem must be identified, and all samples analyzed with the method blank must be reanalyzed.

9.4 Calibration verification checks -- A calibration verification check sample is used to check the accuracy of the instrument and to assess the stability and consistency of the analysis for the analytes of interest. A check sample should be analyzed at the beginning of each working day, during active sample analyses, and at the end of each working day. The frequency of calibration checks during active analysis will depend on the data quality objectives of the project. The check sample should be a well characterized soil sample from the site that is representative of site samples in terms of particle size and degree of homogeneity and that contains contaminants at concentrations near the action levels. If a site-specific sample is not available, then an NIST or other SRM that contains the analytes of interest can be used to verify the accuracy of the instrument. The measured value for each target analyte should be within ± 20 percent (%D) of the true value for the calibration verification check to be acceptable. If a measured value falls outside this range, then the check sample should be reanalyzed. If the value continues to fall outside the acceptance range, the instrument should be recalibrated, and the batch of samples analyzed before the unacceptable calibration verification check must be reanalyzed.

9.5 Precision measurements -- The precision of the method is monitored by analyzing a sample with low, moderate, or high concentrations of target analytes. The frequency of precision measurements will depend on the data quality objectives for the data. A minimum of one precision sample should be run per day. Each precision sample should be analyzed 7 times in replicate. It is recommended that precision measurements be obtained for samples with varying concentration ranges to assess the effect of concentration on method precision. Determining method precision for analytes at concentrations near the site action levels can be extremely important if the FPXRF results are to be used in an enforcement action; therefore, selection of at least one sample with target analyte concentrations at or near the site action levels or levels of concern is recommended. A precision sample is analyzed by the instrument for the same field analysis time as used for other project samples. The relative standard deviation (RSD) of the sample mean is used to assess method precision. For FPXRF data to be considered adequately precise, the RSD should not be greater than 20 percent with the exception of chromium. RSD values for chromium should not be greater than 30 percent. If both in situ and intrusive analytical techniques are used during the course of one day, it is recommended that separate precision calculations be performed for each analysis type.

The equation for calculating RSD is as follows:

$$\text{RSD} = (\text{SD}/\text{Mean Concentration}) \times 100$$

where:

RSD	=	Relative standard deviation for the precision measurement for the analyte
SD	=	Standard deviation of the concentration for the analyte
Mean concentration	=	Mean concentration for the analyte

The precision or reproducibility of a measurement will improve with increasing count time, however, increasing the count time by a factor of 4 will provide only 2 times better precision, so there is a point of diminishing return. Increasing the count time also improves the sensitivity, but decreases sample throughput.

9.6 The lower limits of detection should be established from actual measured performance based on spike recoveries in the matrix of concern or from acceptable method performance on a certified reference material of the appropriate matrix and within the appropriate calibration range for the application. This is considered the best estimate of the true method sensitivity as opposed to a statistical determination based on the standard deviation of

replicate analyses of a low-concentration sample. While the statistical approach demonstrates the potential data variability for a given sample matrix at one point in time, it does not represent what can be detected or most importantly the lowest concentration that can be calibrated. For this reason the sensitivity should be established as the lowest point of detection based on acceptable target analyte recovery in the desired sample matrix.

9.7 Confirmatory samples -- The comparability of the FPXRF analysis is determined by submitting FPXRF-analyzed samples for analysis at a laboratory. The method of confirmatory analysis must meet the project and XRF measurement data quality objectives. The confirmatory samples must be splits of the well homogenized sample material. In some cases the prepared sample cups can be submitted. A minimum of 1 sample for each 20 FPXRF-analyzed samples should be submitted for confirmatory analysis. This frequency will depend on project-specific data quality objectives. The confirmatory analyses can also be used to verify the quality of the FPXRF data. The confirmatory samples should be selected from the lower, middle, and upper range of concentrations measured by the FPXRF. They should also include samples with analyte concentrations at or near the site action levels. The results of the confirmatory analysis and FPXRF analyses should be evaluated with a least squares linear regression analysis. If the measured concentrations span more than one order of magnitude, the data should be log-transformed to standardize variance which is proportional to the magnitude of measurement. The correlation coefficient (r) for the results should be 0.7 or greater for the FPXRF data to be considered screening level data. If the r is 0.9 or greater and inferential statistics indicate the FPXRF data and the confirmatory data are statistically equivalent at a 99 percent confidence level, the data could potentially meet definitive level data criteria.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Instrument calibration -- Instrument calibration procedures vary among FPXRF instruments. Users of this method should follow the calibration procedures outlined in the operator's manual for each specific FPXRF instrument. Generally, however, three types of calibration procedures exist for FPXRF instruments, namely: FP calibration, empirical calibration, and the Compton peak ratio or normalization method. These three types of calibration are discussed below.

10.2 Fundamental parameters calibration -- FP calibration procedures are extremely variable. An FP calibration provides the analyst with a "standardless" calibration. The advantages of FP calibrations over empirical calibrations include the following:

- No previously collected site-specific samples are necessary, although site-specific samples with confirmed and validated analytical results for all elements present could be used.
- Cost is reduced because fewer confirmatory laboratory results or calibration standards are necessary.

However, the analyst should be aware of the limitations imposed on FP calibration by particle size and matrix effects. These limitations can be minimized by adhering to the preparation procedure described in Sec. 7.3. The two FP calibration processes discussed below are based on an effective energy FP routine and a back scatter with FP (BFP) routine. Each FPXRF FP calibration process is based on a different iterative algorithmic method. The calibration procedure for each routine is explained in detail in the manufacturer's user manual for each FPXRF instrument; in addition, training courses are offered for each instrument.

10.2.1 Effective energy FP calibration -- The effective energy FP calibration is performed by the manufacturer before an instrument is sent to the analyst. Although SSCS can be used, the calibration relies on pure element standards or SRMs such as those obtained from NIST for the FP calibration. The effective energy routine relies on the spectrometer response to pure elements and FP iterative algorithms to compensate for various matrix effects.

Alpha coefficients are calculated using a variation of the Sherman equation, which calculates theoretical intensities from the measurement of pure element samples. These coefficients indicate the quantitative effect of each matrix element on an analyte's measured x-ray intensity. Next, the Lachance Traill algorithm is solved as a set of simultaneous equations based on the theoretical intensities. The alpha coefficients are then downloaded into the specific instrument.

The working effective energy FP calibration curve must be verified before sample analysis begins on each working day, after every 20 samples are analyzed, and at the end of sampling. This verification is performed by analyzing either an NIST SRM or an SSCS that is representative of the site-specific samples. This SRM or SSCS serves as a calibration check. A manufacturer-recommended count time per source should be used for the calibration check. The analyst must then adjust the y-intercept and slope of the calibration curve to best fit the known concentrations of target analytes in the SRM or SSCS.

A percent difference (%D) is then calculated for each target analyte. The %D should be within ± 20 percent of the certified value for each analyte. If the %D falls outside this acceptance range, then the calibration curve should be adjusted by varying the slope of the line or the y-intercept value for the analyte. The SRM or SSCS is reanalyzed until the %D falls within ± 20 percent. The group of 20 samples analyzed before an out-of-control calibration check should be reanalyzed.

The equation to calibrate %D is as follows:

$$\%D = ((C_s - C_k) / C_k) \times 100$$

where:

%D = Percent difference

C_k = Certified concentration of standard sample

C_s = Measured concentration of standard sample

10.2.2 BFP calibration -- BFP calibration relies on the ability of the liquid nitrogen-cooled, Si(Li) solid-state detector to separate the coherent (Compton) and incoherent (Rayleigh) backscatter peaks of primary radiation. These peak intensities are known to be a function of sample composition, and the ratio of the Compton to Rayleigh peak is a function of the mass absorption of the sample. The calibration procedure is explained in detail in the instrument manufacturer's manual. Following is a general description of the BFP calibration procedure.

The concentrations of all detected and quantified elements are entered into the computer software system. Certified element results for an NIST SRM or confirmed and validated results for an SSCS can be used. In addition, the concentrations of oxygen and silicon must be entered; these two concentrations are not found in standard metals analyses. The manufacturer provides silicon and oxygen concentrations for typical soil types. Pure element standards are then analyzed using a manufacturer-recommended

count time per source. The results are used to calculate correction factors in order to adjust for spectrum overlap of elements.

The working BFP calibration curve must be verified before sample analysis begins on each working day, after every 20 samples are analyzed, and at the end of the analysis. This verification is performed by analyzing either an NIST SRM or an SSCS that is representative of the site-specific samples. This SRM or SSCS serves as a calibration check. The standard sample is analyzed using a manufacturer-recommended count time per source to check the calibration curve. The analyst must then adjust the y-intercept and slope of the calibration curve to best fit the known concentrations of target analytes in the SRM or SSCS.

A %D is then calculated for each target analyte. The %D should fall within ± 20 percent of the certified value for each analyte. If the %D falls outside this acceptance range, then the calibration curve should be adjusted by varying the slope of the line the y-intercept value for the analyte. The standard sample is reanalyzed until the %D falls within ± 20 percent. The group of 20 samples analyzed before an out-of-control calibration check should be reanalyzed.

10.3 Empirical calibration -- An empirical calibration can be performed with SSCS, site-typical standards, or standards prepared from metal oxides. A discussion of SSCS is included in Sec. 7.3; if no previously characterized samples exist for a specific site, site-typical standards can be used. Site-typical standards may be selected from commercially available characterized soils or from SSCS prepared for another site. The site-typical standards should closely approximate the site's soil matrix with respect to particle size distribution, mineralogy, and contaminant analytes. If neither SSCS nor site-typical standards are available, it is possible to make gravimetric standards by adding metal oxides to a "clean" sand or silicon dioxide matrix that simulates soil. Metal oxides can be purchased from various chemical vendors. If standards are made on site, a balance capable of weighing items to at least two decimal places is necessary. Concentrated ICP or AA standard solutions can also be used to make standards. These solutions are available in concentrations of 10,000 parts per million, thus only small volumes have to be added to the soil.

An empirical calibration using SSCS involves analysis of SSCS by the FPXRF instrument and by a conventional analytical method such as ICP or AA. A total acid digestion procedure should be used by the laboratory for sample preparation. Generally, a minimum of 10 and a maximum of 30 well characterized SSCS, site-typical standards, or prepared metal oxide standards are necessary to perform an adequate empirical calibration. The exact number of standards depends on the number of analytes of interest and interfering elements. Theoretically, an empirical calibration with SSCS should provide the most accurate data for a site because the calibration compensates for site-specific matrix effects.

The first step in an empirical calibration is to analyze the pure element standards for the elements of interest. This enables the instrument to set channel limits for each element for spectral deconvolution. Next the SSCS, site-typical standards, or prepared metal oxide standards are analyzed using a count time of 200 seconds per source or a count time recommended by the manufacturer. This will produce a spectrum and net intensity of each analyte in each standard. The analyte concentrations for each standard are then entered into the instrument software; these concentrations are those obtained from the laboratory, the certified results, or the gravimetrically determined concentrations of the prepared standards. This gives the instrument analyte values to regress against corresponding intensities during the modeling stage. The regression equation correlates the concentrations of an analyte with its net intensity.

The calibration equation is developed using a least squares fit regression analysis. After the regression terms to be used in the equation are defined, a mathematical equation can be developed to calculate the analyte concentration in an unknown sample. In some FPXRF instruments, the software of the instrument calculates the regression equation. The software uses calculated intercept and slope values to form a multiterm equation. In conjunction with the software in the instrument, the operator can adjust the multiterm equation to minimize interelement interferences and optimize the intensity calibration curve.

It is possible to define up to six linear or nonlinear terms in the regression equation. Terms can be added and deleted to optimize the equation. The goal is to produce an equation with the smallest regression error and the highest correlation coefficient. These values are automatically computed by the software as the regression terms are added, deleted, or modified. It is also possible to delete data points from the regression line if these points are significant outliers or if they are heavily weighing the data. Once the regression equation has been selected for an analyte, the equation can be entered into the software for quantitation of analytes in subsequent samples. For an empirical calibration to be acceptable, the regression equation for a specific analyte should have a correlation coefficient of 0.98 or greater or meet the DQOs of the project.

In an empirical calibration, one must apply the DQOs of the project and ascertain critical or action levels for the analytes of interest. It is within these concentration ranges or around these action levels that the FPXRF instrument should be calibrated most accurately. It may not be possible to develop a good regression equation over several orders of analyte concentration.

10.4 Compton normalization method -- The Compton normalization method is based on analysis of a single, certified standard and normalization for the Compton peak. The Compton peak is produced from incoherent backscattering of x-ray radiation from the excitation source and is present in the spectrum of every sample. The Compton peak intensity changes with differing matrices. Generally, matrices dominated by lighter elements produce a larger Compton peak, and those dominated by heavier elements produce a smaller Compton peak. Normalizing to the Compton peak can reduce problems with varying matrix effects among samples. Compton normalization is similar to the use of internal standards in organics analysis. The Compton normalization method may not be effective when analyte concentrations exceed a few percent.

The certified standard used for this type of calibration could be an NIST SRM such as 2710 or 2711. The SRM must be a matrix similar to the samples and must contain the analytes of interests at concentrations near those expected in the samples. First, a response factor has to be determined for each analyte. This factor is calculated by dividing the net peak intensity by the analyte concentration. The net peak intensity is gross intensity corrected for baseline reading. Concentrations of analytes in samples are then determined by multiplying the baseline corrected analyte signal intensity by the normalization factor and by the response factor. The normalization factor is the quotient of the baseline corrected Compton K_{α} peak intensity of the SRM divided by that of the samples. Depending on the FPXRF instrument used, these calculations may be done manually or by the instrument software.

11.0 PROCEDURE

11.1 Operation of the various FPXRF instruments will vary according to the manufacturers' protocols. Before operating any FPXRF instrument, one should consult the manufacturer's manual. Most manufacturers recommend that their instruments be allowed to warm up for 15 to 30 minutes before analysis of samples. This will help alleviate drift or energy calibration problems later during analysis.

11.2 Each FPXRF instrument should be operated according to the manufacturer's recommendations. There are two modes in which FPXRF instruments can be operated: in situ and intrusive. The in situ mode involves analysis of an undisturbed soil sediment or sample. Intrusive analysis involves collection and preparation of a soil or sediment sample before analysis. Some FPXRF instruments can operate in both modes of analysis, while others are designed to operate in only one mode. The two modes of analysis are discussed below.

11.3 For in situ analysis, remove any large or nonrepresentative debris from the soil surface before analysis. This debris includes rocks, pebbles, leaves, vegetation, roots, and concrete. Also, the soil surface must be as smooth as possible so that the probe window will have good contact with the surface. This may require some leveling of the surface with a stainless-steel trowel. During the study conducted to provide example performance data for this method, this modest amount of sample preparation was found to take less than 5 min per sample location. The last requirement is that the soil or sediment not be saturated with water. Manufacturers state that their FPXRF instruments will perform adequately for soils with moisture contents of 5 to 20 percent but will not perform well for saturated soils, especially if ponded water exists on the surface. Another recommended technique for in situ analysis is to tamp the soil to increase soil density and compactness for better repeatability and representativeness. This condition is especially important for heavy element analysis, such as barium. Source count times for in situ analysis usually range from 30 to 120 seconds, but source count times will vary among instruments and depending on the desired method sensitivity. Due to the heterogeneous nature of the soil sample, in situ analysis can provide only "screening" type data.

11.4 For intrusive analysis of surface or sediment, it is recommended that a sample be collected from a 4- by 4-inch square that is 1 inch deep. This will produce a soil sample of approximately 375 g or 250 cm³, which is enough soil to fill an 8-ounce jar. However, the exact dimensions and sample depth should take into consideration the heterogeneous deposition of contaminants and will ultimately depend on the desired project-specific data quality objectives. The sample should be homogenized, dried, and ground before analysis. The sample can be homogenized before or after drying. The homogenization technique to be used after drying is discussed in Sec. 4.2. If the sample is homogenized before drying, it should be thoroughly mixed in a beaker or similar container, or if the sample is moist and has a high clay content, it can be kneaded in a plastic bag. One way to monitor homogenization when the sample is kneaded in a plastic bag is to add sodium fluorescein dye to the sample. After the moist sample has been homogenized, it is examined under an ultraviolet light to assess the distribution of sodium fluorescein throughout the sample. If the fluorescent dye is evenly distributed in the sample, homogenization is considered complete; if the dye is not evenly distributed, mixing should continue until the sample has been thoroughly homogenized. During the study conducted to provide data for this method, the time necessary for homogenization procedure using the fluorescein dye ranged from 3 to 5 min per sample. As demonstrated in Secs. 13.5 and 13.7, homogenization has the greatest impact on the reduction of sampling variability. It produces little or no contamination. Often, the direct analysis through the plastic bag is possible without the more labor intensive steps of drying, grinding, and sieving given in Secs. 11.5 and 11.6. Of course, to achieve the best data quality possible all four steps should be followed.

11.5 Once the soil or sediment sample has been homogenized, it should be dried. This can be accomplished with a toaster oven or convection oven. A small aliquot of the sample (20 to 50 g) is placed in a suitable container for drying. The sample should be dried for 2 to 4 hr in the convection or toaster oven at a temperature not greater than 150 °C. Samples may also be air dried under ambient temperature conditions using a 10- to 20-g portion. Regardless of what drying mechanism is used, the drying process is considered complete when a constant sample weight can be obtained. Care should be taken to avoid sample cross-contamination and these measures can be evaluated by including an appropriate method blank sample along with any sample preparation process.

CAUTION: Microwave drying is not a recommended procedure. Field studies have shown that microwave drying can increase variability between the FPXRF data and confirmatory analysis. High levels of metals in a sample can cause arcing in the microwave oven, and sometimes slag forms in the sample. Microwave oven drying can also melt plastic containers used to hold the sample.

11.6 The homogenized dried sample material should be ground with a mortar and pestle and passed through a 60-mesh sieve to achieve a uniform particle size. Sample grinding should continue until at least 90 percent of the original sample passes through the sieve. The grinding step normally takes an average of 10 min per sample. An aliquot of the sieved sample should then be placed in a 31.0-mm polyethylene sample cup (or equivalent) for analysis. The sample cup should be one-half to three-quarters full at a minimum. The sample cup should be covered with a 2.5 μm Mylar (or equivalent) film for analysis. The rest of the soil sample should be placed in a jar, labeled, and archived for possible confirmation analysis. All equipment including the mortar, pestle, and sieves must be thoroughly cleaned so that any cross-contamination is below the established lower limit of detection of the procedure or DQOs of the analysis. If all recommended sample preparation steps are followed, there is a high probability the desired laboratory data quality may be obtained.

12.0 DATA ANALYSIS AND CALCULATIONS

Most FPXRF instruments have software capable of storing all analytical results and spectra. The results are displayed in ppm and can be downloaded to a personal computer, which can be used to provide a hard copy printout. Individual measurements that are smaller than three times their associated SD should not be used for quantitation. See the manufacturer's instructions regarding data analysis and calculations.

13.0 METHOD PERFORMANCE

13.1 Performance data and related information are provided in SW-846 methods only as examples and guidance. The data do not represent required performance criteria for users of the methods. Instead, performance criteria should be developed on a project-specific basis, and the laboratory should establish in-house QC performance criteria for the application of this method. These performance data are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

13.2 The sections to follow discuss three performance evaluation factors; namely, precision, accuracy, and comparability. The example data presented in Tables 4 through 8 were generated from results obtained from six FPXRF instruments (see Sec. 13.3). The soil samples analyzed by the six FPXRF instruments were collected from two sites in the United States. The soil samples contained several of the target analytes at concentrations ranging from "nondetect" to tens of thousands of mg/kg. These data are provided for guidance purposes only.

13.3 The six FPXRF instruments included the TN 9000 and TN Lead Analyzer manufactured by TN Spectrace; the X-MET 920 with a SiLi detector and X-MET 920 with a gas-filled proportional detector manufactured by Metorex, Inc.; the XL Spectrum Analyzer manufactured by Niton; and the MAP Spectrum Analyzer manufactured by Scitec. The TN 9000 and TN Lead Analyzer both have a HgI_2 detector. The TN 9000 utilized an Fe-55, Cd-109, and Am-241 source. The TN Lead Analyzer had only a Cd-109 source. The X-Met 920 with the SiLi detector had a Cd-109 and Am-241 source. The X-MET 920 with the gas-filled proportional detector had only a Cd-109 source. The XL Spectrum Analyzer utilized a silicon pin-diode

detector and a Cd-109 source. The MAP Spectrum Analyzer utilized a solid-state silicon detector and a Cd-109 source.

13.4 All example data presented in Tables 4 through 8 were generated using the following calibrations and source count times. The TN 9000 and TN Lead Analyzer were calibrated using fundamental parameters using NIST SRM 2710 as a calibration check sample. The TN 9000 was operated using 100, 60, and 60 second count times for the Cd-109, Fe-55, and Am-241 sources, respectively. The TN Lead analyzer was operated using a 60 second count time for the Cd-109 source. The X-MET 920 with the Si(Li) detector was calibrated using fundamental parameters and one well characterized site-specific soil standard as a calibration check. It used 140 and 100 second count times for the Cd-109 and Am-241 sources, respectively. The X-MET 920 with the gas-filled proportional detector was calibrated empirically using between 10 and 20 well characterized site-specific soil standards. It used 120 second times for the Cd-109 source. The XL Spectrum Analyzer utilized NIST SRM 2710 for calibration and the Compton peak normalization procedure for quantitation based on 60 second count times for the Cd-109 source. The MAP Spectrum Analyzer was internally calibrated by the manufacturer. The calibration was checked using a well-characterized site-specific soil standard. It used 240 second times for the Cd-109 source.

13.5 Precision measurements -- The example precision data are presented in Table 4. These data are provided for guidance purposes only. Each of the six FPXRF instruments performed 10 replicate measurements on 12 soil samples that had analyte concentrations ranging from "nondetects" to thousands of mg/kg. Each of the 12 soil samples underwent 4 different preparation techniques from in situ (no preparation) to dried and ground in a sample cup. Therefore, there were 48 precision data points for five of the instruments and 24 precision points for the MAP Spectrum Analyzer. The replicate measurements were taken using the source count times discussed at the beginning of this section.

For each detectable analyte in each precision sample a mean concentration, standard deviation, and RSD was calculated for each analyte. The data presented in Table 4 is an average RSD for the precision samples that had analyte concentrations at 5 to 10 times the lower limit of detection for that analyte for each instrument. Some analytes such as mercury, selenium, silver, and thorium were not detected in any of the precision samples so these analytes are not listed in Table 4. Some analytes such as cadmium, nickel, and tin were only detected at concentrations near the lower limit of detection so that an RSD value calculated at 5 to 10 times this limit was not possible.

One FPXRF instrument collected replicate measurements on an additional nine soil samples to provide a better assessment of the effect of sample preparation on precision. Table 5 shows these results. These data are provided for guidance purposes only. The additional nine soil samples were comprised of three from each texture and had analyte concentrations ranging from near the lower limit of detection for the FPXRF analyzer to thousands of mg/kg. The FPXRF analyzer only collected replicate measurements from three of the preparation methods; no measurements were collected from the in situ homogenized samples. The FPXRF analyzer conducted five replicate measurements of the in situ field samples by taking measurements at five different points within the 4-inch by 4-inch sample square. Ten replicate measurements were collected for both the intrusive undried and unground and intrusive dried and ground samples contained in cups. The cups were shaken between each replicate measurement.

Table 5 shows that the precision dramatically improved from the in situ to the intrusive measurements. In general there was a slight improvement in precision when the sample was dried and ground. Two factors caused the precision for the in situ measurements to be poorer. The major factor is soil heterogeneity. By moving the probe within the 4-inch by 4-inch square,

measurements of different soil samples were actually taking place within the square. Table 5 illustrates the dominant effect of soil heterogeneity. It overwhelmed instrument precision when the FPXRF analyzer was used in this mode. The second factor that caused the RSD values to be higher for the in situ measurements is the fact that only five instead of ten replicates were taken. A lesser number of measurements caused the standard deviation to be larger which in turn elevated the RSD values.

13.6 Accuracy measurements -- Five of the FPXRF instruments (not including the MAP Spectrum Analyzer) analyzed 18 SRMs using the source count times and calibration methods given at the beginning of this section. The 18 SRMs included 9 soil SRMs, 4 stream or river sediment SRMs, 2 sludge SRMs, and 3 ash SRMs. Each of the SRMs contained known concentrations of certain target analytes. A percent recovery was calculated for each analyte in each SRM for each FPXRF instrument. Table 6 presents a summary of this data. With the exception of cadmium, chromium, and nickel, the values presented in Table 6 were generated from the 13 soil and sediment SRMs only. The 2 sludge and 3 ash SRMs were included for cadmium, chromium, and nickel because of the low or nondetectable concentrations of these three analytes in the soil and sediment SRMs.

Only 12 analytes are presented in Table 6. These are the analytes that are of environmental concern and provided a significant number of detections in the SRMs for an accuracy assessment. No data is presented for the X-MET 920 with the gas-filled proportional detector. This FPXRF instrument was calibrated empirically using site-specific soil samples. The percent recovery values from this instrument were very sporadic and the data did not lend itself to presentation in Table 6.

Table 7 provides a more detailed summary of accuracy data for one particular FPXRF instrument (TN 9000) for the 9 soil SRMs and 4 sediment SRMs. These data are provided for guidance purposes only. Table 7 shows the certified value, measured value, and percent recovery for five analytes. These analytes were chosen because they are of environmental concern and were most prevalently certified for in the SRM and detected by the FPXRF instrument. The first nine SRMs are soil and the last 4 SRMs are sediment. Percent recoveries for the four NIST SRMs were often between 90 and 110 percent for all analytes.

13.7 Comparability -- Comparability refers to the confidence with which one data set can be compared to another. In this case, FPXRF data generated from a large study of six FPXRF instruments was compared to SW-846 Methods 3050 and 6010 which are the standard soil extraction for metals and analysis by inductively coupled plasma. An evaluation of comparability was conducted by using linear regression analysis. Three factors were determined using the linear regression. These factors were the y-intercept, the slope of the line, and the coefficient of determination (r^2).

As part of the comparability assessment, the effects of soil type and preparation methods were studied. Three soil types (textures) and four preparation methods were examined during the study. The preparation methods evaluated the cumulative effect of particle size, moisture, and homogenization on comparability. Due to the large volume of data produced during this study, linear regression data for six analytes from only one FPXRF instrument is presented in Table 8. Similar trends in the data were seen for all instruments. These data are provided for guidance purposes only.

Table 8 shows the regression parameters for the whole data set, broken out by soil type, and by preparation method. These data are provided for guidance purposes only. The soil types are as follows: soil 1--sand; soil 2--loam; and soil 3--silty clay. The preparation methods are as follows: preparation 1--in situ in the field; preparation 2--intrusive, sample collected and homogenized; preparation 3--intrusive, with sample in a sample cup but sample still wet and not

ground; and preparation 4—intrusive, with sample dried, ground, passed through a 40-mesh sieve, and placed in sample cup.

For arsenic, copper, lead, and zinc, the comparability to the confirmatory laboratory was excellent with r^2 values ranging from 0.80 to 0.99 for all six FPXRF instruments. The slopes of the regression lines for arsenic, copper, lead, and zinc, were generally between 0.90 and 1.00 indicating the data would need to be corrected very little or not at all to match the confirmatory laboratory data. The r^2 values and slopes of the regression lines for barium and chromium were not as good as for the other for analytes, indicating the data would have to be corrected to match the confirmatory laboratory.

Table 8 demonstrates that there was little effect of soil type on the regression parameters for any of the six analytes. The only exceptions were for barium in soil 1 and copper in soil 3. In both of these cases, however, it is actually a concentration effect and not a soil effect causing the poorer comparability. All barium and copper concentrations in soil 1 and 3, respectively, were less than 350 mg/kg.

Table 8 shows there was a preparation effect on the regression parameters for all six analytes. With the exception of chromium, the regression parameters were primarily improved going from preparation 1 to preparation 2. In this step, the sample was removed from the soil surface, all large debris was removed, and the sample was thoroughly homogenized. The additional two preparation methods did little to improve the regression parameters. This data indicates that homogenization is the most critical factor when comparing the results. It is essential that the sample sent to the confirmatory laboratory match the FPXRF sample as closely as possible.

Sec. 11.0 of this method discusses the time necessary for each of the sample preparation techniques. Based on the data quality objectives for the project, an analyst must decide if it is worth the extra time necessary to dry and grind the sample for small improvements in comparability. Homogenization requires 3 to 5 min. Drying the sample requires one to two hours. Grinding and sieving requires another 10 to 15 min per sample. Lastly, when grinding and sieving is conducted, time has to be allotted to decontaminate the mortars, pestles, and sieves. Drying and grinding the samples and decontamination procedures will often dictate that an extra person be on site so that the analyst can keep up with the sample collection crew. The cost of requiring an extra person on site to prepare samples must be balanced with the gain in data quality and sample throughput.

13.8 The following documents may provide additional guidance and insight on this method and technique:

13.8.1 A. D. Hewitt, "Screening for Metals by X-ray Fluorescence Spectrometry/Response Factor/Compton K_α Peak Normalization Analysis," American Environmental Laboratory, pp 24-32, 1994.

13.8.2 S. Piorek and J. R. Pasmore, "Standardless, In Situ Analysis of Metallic Contaminants in the Natural Environment With a PC-Based, High Resolution Portable X-Ray Analyzer," Third International Symposium on Field Screening Methods for Hazardous Waste and Toxic Chemicals, Las Vegas, Nevada, February 24-26, 1993, Vol 2, pp 1135-1151, 1993.

13.8.3 S. Shefsky, "Sample Handling Strategies for Accurate Lead-in-soil Measurements in the Field and Laboratory," *International Symposium of Field Screening Methods for Hazardous Waste and Toxic Chemicals*, Las Vegas, NV, January 29-31, 1997.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical Management for Waste Reduction* available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th St., N.W. Washington, D.C. 20036, <http://www.acs.org>.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Sec. 14.2.

16.0 REFERENCES

1. Metorex, X-MET 920 User's Manual.
2. Spectrace Instruments, "Energy Dispersive X-ray Fluorescence Spectrometry: An Introduction," 1994.
3. TN Spectrace, Spectrace 9000 Field Portable/Benchtop XRF Training and Applications Manual.
4. Unpublished SITE data, received from PRC Environment Management, Inc.

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

The following pages contain the tables referenced by this method. A flow diagram of the procedure follows the tables.

TABLE 1

EXAMPLE INTERFERENCE FREE LOWER LIMITS OF DETECTION

Analyte	Chemical Abstract Series Number	Lower Limit of Detection in Quartz Sand (milligrams per kilogram)
Antimony (Sb)	7440-36-0	40
Arsenic (As)	7440-38-0	40
Barium (Ba)	7440-39-3	20
Cadmium (Cd)	7440-43-9	100
Calcium (Ca)	7440-70-2	70
Chromium (Cr)	7440-47-3	150
Cobalt (Co)	7440-48-4	60
Copper (Cu)	7440-50-8	50
Iron (Fe)	7439-89-6	60
Lead (Pb)	7439-92-1	20
Manganese (Mn)	7439-96-5	70
Mercury (Hg)	7439-97-6	30
Molybdenum (Mo)	7439-93-7	10
Nickel (Ni)	7440-02-0	50
Potassium (K)	7440-09-7	200
Rubidium (Rb)	7440-17-7	10
Selenium (Se)	7782-49-2	40
Silver (Ag)	7440-22-4	70
Strontium (Sr)	7440-24-6	10
Thallium (Tl)	7440-28-0	20
Thorium (Th)	7440-29-1	10
Tin (Sn)	7440-31-5	60
Titanium (Ti)	7440-32-6	50
Vanadium (V)	7440-62-2	50
Zinc (Zn)	7440-66-6	50
Zirconium (Zr)	7440-67-7	10

Source: Refs. 1, 2, and 3

These data are provided for guidance purposes only.

TABLE 2
SUMMARY OF RADIOISOTOPE SOURCE CHARACTERISTICS

Source	Activity (mCi)	Half-Life (Years)	Excitation Energy (keV)	Elemental Analysis Range	
Fe-55	20-50	2.7	5.9	Sulfur to Chromium Molybdenum to Barium	K Lines L Lines
Cd-109	5-30	1.3	22.1 and 87.9	Calcium to Rhodium Tantalum to Lead Barium to Uranium	K Lines K Lines L Lines
Am-241	5-30	432	26.4 and 59.6	Copper to Thulium Tungsten to Uranium	K Lines L Lines
Cm-244	60-100	17.8	14.2	Titanium to Selenium Lanthanum to Lead	K Lines L Lines

Source: Refs. 1, 2, and 3

TABLE 3
SUMMARY OF X-RAY TUBE SOURCE CHARACTERISTICS

Anode Material	Recommended Voltage Range (kV)	K-alpha Emission (keV)	Elemental Analysis Range	
Cu	18-22	8.04	Potassium to Cobalt Silver to Gadolinium	K Lines L Lines
Mo	40-50	17.4	Cobalt to Yttrium Europium to Radon	K Lines L Lines
Ag	50-65	22.1	Zinc to Technicium Ytterbium to Neptunium	K Lines L Lines

Source: Ref. 4

Notes: The sample elements excited are chosen by taking as the lower limit the same ratio of excitation line energy to element absorption edge as in Table 2 (approximately 0.45) and the requirement that the excitation line energy be above the element absorption edge as the upper limit (L2 edges used for L lines). K-beta excitation lines were ignored.

TABLE 4
EXAMPLE PRECISION VALUES

Analyte	Average Relative Standard Deviation for Each Instrument at 5 to 10 Times the Lower Limit of Detection					
	TN 9000	TN Lead Analyzer	X-MET 920 (SiLi Detector)	X-MET 920 (Gas-Filled Detector)	XL Spectrum Analyzer	MAP Spectrum Analyzer
Antimony	6.54	NR	NR	NR	NR	NR
Arsenic	5.33	4.11	3.23	1.91	12.47	6.68
Barium	4.02	NR	3.31	5.91	NR	NR
Cadmium	29.84 ^a	NR	24.80 ^a	NR	NR	NR
Calcium	2.16	NR	NR	NR	NR	NR
Chromium	22.25	25.78	22.72	3.91	30.25	NR
Cobalt	33.90	NR	NR	NR	NR	NR
Copper	7.03	9.11	8.49	9.12	12.77	14.86
Iron	1.78	1.67	1.55	NR	2.30	NR
Lead	6.45	5.93	5.05	7.56	6.97	12.16
Manganese	27.04	24.75	NR	NR	NR	NR
Molybdenum	6.95	NR	NR	NR	12.60	NR
Nickel	30.85 ^a	NR	24.92 ^a	20.92 ^a	NA	NR
Potassium	3.90	NR	NR	NR	NR	NR
Rubidium	13.06	NR	NR	NR	32.69 ^a	NR
Strontium	4.28	NR	NR	NR	8.86	NR
Tin	24.32 ^a	NR	NR	NR	NR	NR
Titanium	4.87	NR	NR	NR	NR	NR
Zinc	7.27	7.48	4.26	2.28	10.95	0.83
Zirconium	3.58	NR	NR	NR	6.49	NR

These data are provided for guidance purposes only.

Source: Ref. 4

^a These values are biased high because the concentration of these analytes in the soil samples was near the lower limit of detection for that particular FPXRF instrument.

NR Not reported.

NA Not applicable; analyte was reported but was below the established lower limit detection.

TABLE 5

EXAMPLES OF PRECISION AS AFFECTED BY SAMPLE PREPARATION

Analyte	Average Relative Standard Deviation for Each Preparation Method		
	In Situ-Field	Intrusive-Undried and Unground	Intrusive-Dried and Ground
Antimony	30.1	15.0	14.4
Arsenic	22.5	5.36	3.76
Barium	17.3	3.38	2.90
Cadmium ^a	41.2	30.8	28.3
Calcium	17.5	1.68	1.24
Chromium	17.6	28.5	21.9
Cobalt	28.4	31.1	28.4
Copper	26.4	10.2	7.90
Iron	10.3	1.67	1.57
Lead	25.1	8.55	6.03
Manganese	40.5	12.3	13.0
Mercury	ND	ND	ND
Molybdenum	21.6	20.1	19.2
Nickel ^a	29.8	20.4	18.2
Potassium	18.6	3.04	2.57
Rubidium	29.8	16.2	18.9
Selenium	ND	20.2	19.5
Silver ^a	31.9	31.0	29.2
Strontium	15.2	3.38	3.98
Thallium	39.0	16.0	19.5
Thorium	NR	NR	NR
Tin	ND	14.1	15.3
Titanium	13.3	4.15	3.74
Vanadium	NR	NR	NR
Zinc	26.6	13.3	11.1
Zirconium	20.2	5.63	5.18

These data are provided for guidance purposes only.

Source: Ref. 4

^a These values may be biased high because the concentration of these analytes in the soil samples was near the lower limit of detection.

ND Not detected.

NR Not reported.

TABLE 6
EXAMPLE ACCURACY VALUES

Analyte	Instrument															
	TN 9000				TN Lead Analyzer				X-MET 920 (SiLi Detector)				XL Spectrum Analyzer			
	n	Range of % Rec.	Mean % Rec.	SD	n	Range of % Rec.	Mean % Rec.	SD	n	Range of % Rec.	Mean % Rec.	SD	n	Range of % Rec.	Mean % Rec.	SD
Sb	2	100-149	124.3	NA	--	--	--	--	--	--	--	--	--	--	--	--
As	5	68-115	92.8	17.3	5	44-105	83.4	23.2	4	9.7-91	47.7	39.7	5	38-535	189.8	206
Ba	9	98-198	135.3	36.9	--	--	--	--	9	18-848	168.2	262	--	--	--	--
Cd	2	99-129	114.3	NA	--	--	--	--	6	81-202	110.5	45.7	--	--	--	--
Cr	2	99-178	138.4	NA	--	--	--	--	7	22-273	143.1	93.8	3	98-625	279.2	300
Cu	8	61-140	95.0	28.8	6	38-107	79.1	27.0	11	10-210	111.8	72.1	8	95-480	203.0	147
Fe	6	78-155	103.7	26.1	6	89-159	102.3	28.6	6	48-94	80.4	16.2	6	26-187	108.6	52.9
Pb	11	66-138	98.9	19.2	11	68-131	97.4	18.4	12	23-94	72.7	20.9	13	80-234	107.3	39.9
Mn	4	81-104	93.1	9.70	3	92-152	113.1	33.8	--	--	--	--	--	--	--	--
Ni	3	99-122	109.8	12.0	--	--	--	--	--	--	--	--	3	57-123	87.5	33.5
Sr	8	110-178	132.6	23.8	--	--	--	--	--	--	--	--	7	86-209	125.1	39.5
Zn	11	41-130	94.3	24.0	10	81-133	100.0	19.7	12	46-181	106.6	34.7	11	31-199	94.6	42.5

Source: Ref. 4. These data are provided for guidance purposes only.

n: Number of samples that contained a certified value for the analyte and produced a detectable concentration from the FPXRF instrument.

SD: Standard deviation; NA: Not applicable; only two data points, therefore, a SD was not calculated.

%Rec.: Percent recovery.

-- No data.

TABLE 7
EXAMPLE ACCURACY FOR TN 9000^a

Standard Reference Material	Arsenic			Barium			Copper			Lead			Zinc		
	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.
RTC CRM-021	24.8	ND	NA	586	1135	193.5	4792	2908	60.7	144742	149947	103.6	546	224	40.9
RTC CRM-020	397	429	92.5	22.3	ND	NA	753	583	77.4	5195	3444	66.3	3022	3916	129.6
BCR CRM 143R	--	--	--	--	--	--	131	105	80.5	180	206	114.8	1055	1043	99.0
BCR CRM 141	--	--	--	--	--	--	32.6	ND	NA	29.4	ND	NA	81.3	ND	NA
USGS GXR-2	25.0	ND	NA	2240	2946	131.5	76.0	106	140.2	690	742	107.6	530	596	112.4
USGS GXR-6	330	294	88.9	1300	2581	198.5	66.0	ND	NA	101	80.9	80.1	118	ND	NA
NIST 2711	105	104	99.3	726	801	110.3	114	ND	NA	1162	1172	100.9	350	333	94.9
NIST 2710	626	722	115.4	707	782	110.6	2950	2834	96.1	5532	5420	98.0	6952	6476	93.2
NIST 2709	17.7	ND	NA	968	950	98.1	34.6	ND	NA	18.9	ND	NA	106	98.5	93.0
NIST 2704	23.4	ND	NA	414	443	107.0	98.6	105	106.2	161	167	103.5	438	427	97.4
CNRC PACS-1	211	143	67.7	--	772	NA	452	302	66.9	404	332	82.3	824	611	74.2
SARM-51	--	--	--	335	466	139.1	268	373	139.2	5200	7199	138.4	2200	2676	121.6
SARM-52	--	--	--	410	527	128.5	219	193	88.1	1200	1107	92.2	264	215	81.4

Source: Ref. 4. These data are provided for guidance purposes only.

^a All concentrations in milligrams per kilogram.

%Rec.: Percent recovery; ND: Not detected; NA: Not applicable.

-- No data.

TABLE 8

EXAMPLE REGRESSION PARAMETERS FOR COMPARABILITY¹

	Arsenic				Barium				Copper			
	n	r ²	Int.	Slope	n	r ²	Int.	Slope	n	r ²	Int.	Slope
All Data	824	0.94	1.62	0.94	1255	0.71	60.3	0.54	984	0.93	2.19	0.93
Soil 1	368	0.96	1.41	0.95	393	0.05	42.6	0.11	385	0.94	1.26	0.99
Soil 2	453	0.94	1.51	0.96	462	0.56	30.2	0.66	463	0.92	2.09	0.95
Soil 3	—	—	—	—	400	0.85	44.7	0.59	136	0.46	16.60	0.57
Prep 1	207	0.87	2.69	0.85	312	0.64	53.7	0.55	256	0.87	3.89	0.87
Prep 2	208	0.97	1.38	0.95	315	0.67	64.6	0.52	246	0.96	2.04	0.93
Prep 3	204	0.96	1.20	0.99	315	0.78	64.6	0.53	236	0.97	1.45	0.99
Prep 4	205	0.96	1.45	0.98	313	0.81	58.9	0.55	246	0.96	1.99	0.96

	Lead				Zinc				Chromium			
	n	r ²	Int.	Slope	n	r ²	Int.	Slope	n	r ²	Int.	Slope
All Data	1205	0.92	1.66	0.95	1103	0.89	1.86	0.95	280	0.70	64.6	0.42
Soil 1	357	0.94	1.41	0.96	329	0.93	1.78	0.93	—	—	—	—
Soil 2	451	0.93	1.62	0.97	423	0.85	2.57	0.90	—	—	—	—
Soil 3	397	0.90	2.40	0.90	351	0.90	1.70	0.98	186	0.66	38.9	0.50
Prep 1	305	0.80	2.88	0.86	286	0.79	3.16	0.87	105	0.80	66.1	0.43
Prep 2	298	0.97	1.41	0.96	272	0.95	1.86	0.93	77	0.51	81.3	0.36
Prep 3	302	0.98	1.26	0.99	274	0.93	1.32	1.00	49	0.73	53.7	0.45
Prep 4	300	0.96	1.38	1.00	271	0.94	1.41	1.01	49	0.75	31.6	0.56

Source: Ref. 4. These data are provided for guidance purposes only.

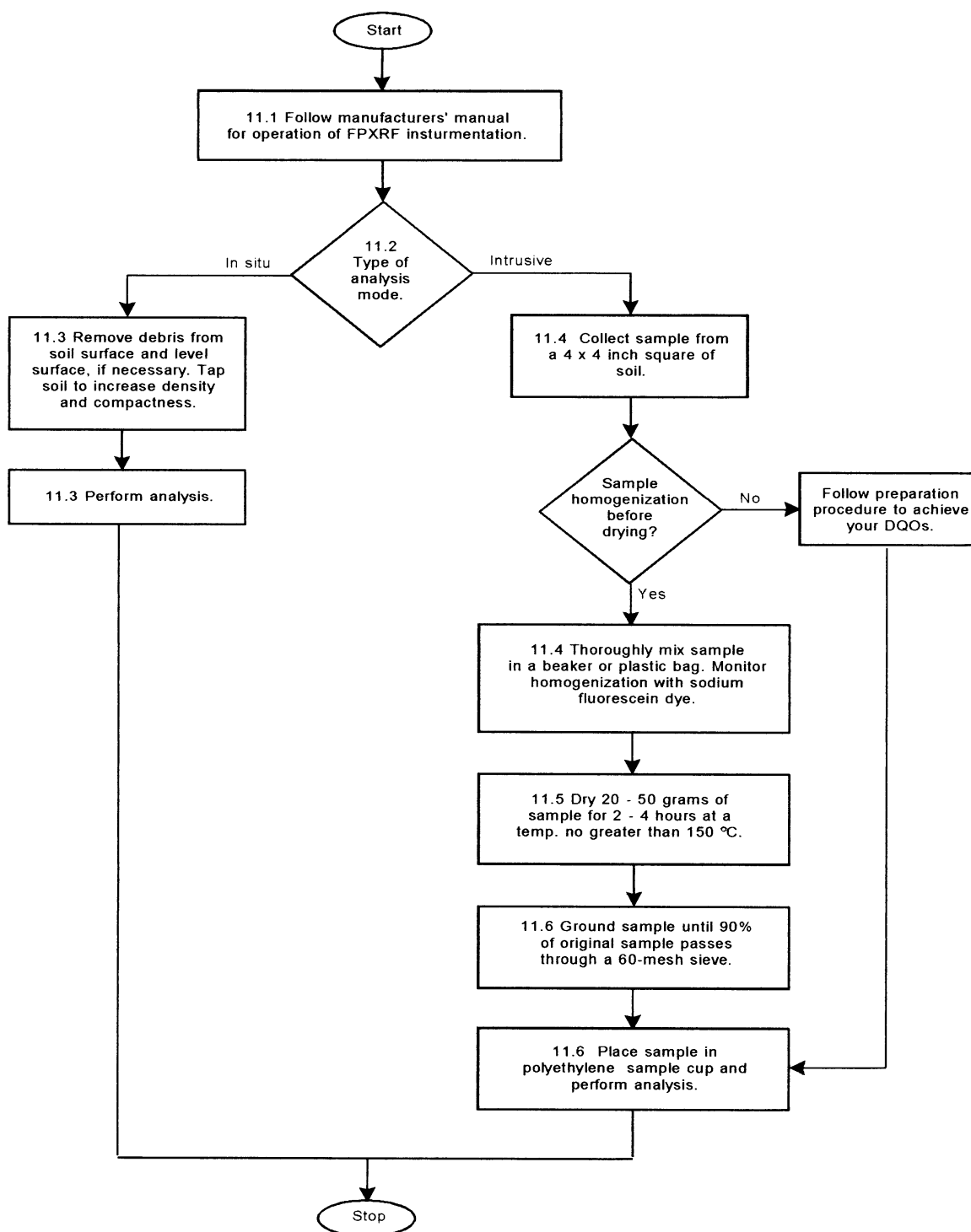
¹ Log-transformed data

n: Number of data points; r²: Coefficient of determination; Int.: Y-intercept

— No applicable data

METHOD 6200

FIELD PORTABLE X-RAY FLUORESCENCE SPECTROMETRY FOR THE DETERMINATION OF ELEMENTAL CONCENTRATIONS IN SOIL AND SEDIMENT





St. Charles, Illinois

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